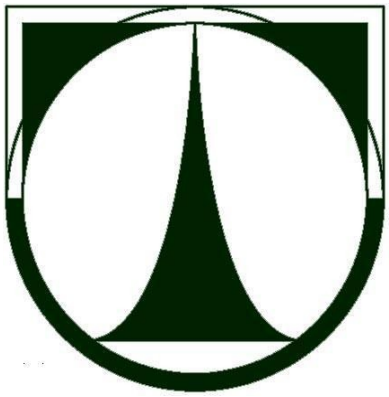
Technical University of Liberec

Faculty of Textile Engineering



DIPLOMA THESIS

2010 Yoliswa Sidloyi

Technical University of Liberec

Faculty of Textile Engineering

Department of Textile Chemistry

Flame retardant finish on furniture textiles

Yoliswa Sidloyi

Supervisor: Assoc. Prof. Miroslav Prášil.

Consultant: Assoc. Prof. Jakub Wiener, PhD.

Number of text pages: 123

Number of pictures: 35

Number of tables: 17

Number of graphs: 22

Statement

I have been informed that on my thesis is fully applicable the Act No. 121/2000 Coll. about copyright, especially §60 - school work.

I acknowledge that Technical University of Liberec (TUL) does not breach my copyright when using my thesis for internal need of TUL.

Shall I use my thesis or shall I award a licence for its utilisation I acknowledge that I am obliged to inform TUL about this fact, TUL has right to claim expenses incurred for this thesis up to amount of actual full expenses.

I have elaborate the thesis alone utilising listed and on basis of consultations with supervisor.

Date: 12 May 2010

Signature: Yoliswa Sidloyi

Acknowledgements

I would like to take this opportunity to express my gratitude to my supervisor Assoc. Prof. Miroslav Prášil for supervising me, providing resources and subjects, and offering direction and penetrating criticism; Assoc. Prof. Jakob Wiener for passing me many leads, and helping to set up the diploma thesis.

I would like to thank Inotex spol. s r.o., Dvůr Králové n/L by giving me an opportunity to do my practicals in their laboratories and assistance of Ing. Lenka Martínková. A special thanks to University of Liberec and the Faculty of Textile engineering laboratories for letting me use their laboratories and providing documents for studying.

I cannot end without thanking my family, on whose constant encouragement and love I have relied throughout my time at the Academy. I am grateful also to the examples of my late grandmother, Nomsebenzi Mary-Jane Sidloyi and my mother Nontozamo Constance Sidloyi. Their unflinching courage and conviction will always inspire me, and I hope to continue, in my own small way, the noble mission to which they gave their lives. It is to them that I dedicate this work.

Abstract

Flame retardants save many lives and property because they prevent accidental fires. On the other hand, there are concerns related to chemical release into the environment and potential health effects. Since halogenated flame retardants have been in the focus of public scrutiny, flame retardants based on other chemistries like phosphorus and nitrogen have been developed and need to prove their environmental benefits.

This study is concentrating on discussion of specific problems of flammability and flame retardation of the polymer in terms of its molecular structure and its application. The methodology of testing for polymer flammability, the significance of test results, and their correlation with fire hazard form a complex subject which is beyond the scope of this diploma thesis. Flame retardancy standards for public safety are generally controlled or influenced by government departments. Flame retardancy is required in many coated products, and a polymer coating can hold a larger amount of FR chemical than a simple finish on a fabric.

The use of coatings is widely used and for many purposes. Most items that we own are coated. Coating is applied to render a certain property on a fabric and to protect materials from corrosion and other detrimental effects of the ambient atmosphere. Surface properties such as gloss, color, slipperiness, they change and make the surface beautiful. In many cases coatings are applied to improve surface properties of the substrate, such as appearance, adhesion, wet ability, wear resistance, and scratch resistance. Wide range of applications techniques, coating types and purposes makes coating an extremely diverse field.

Coatings may be applied as liquid, gases or solids. Coatings can be measured and tested for proper opacity and film thickness. There are processes that had to be followed when applying coating on a fabric. Achieving high level properties in coating technical textiles requires implicit knowledge of the fabric, the polymers with their strengths and weaknesses, and the technology. The right selection and combination of polymers depend on end use and technical properties. . Literature is covering various aspects of polymer flammability, flame-retardant compounds for polymers, and of possible improvements in the fire safety.

Contents

Nomenclature

Ac – Acrylic

ATH –Alumina Trihydrate

Br – Bromine

cl – chlorine

CO – Cotton

decaBDE – decabromodiphenyl ether

Eq – Equation

FR – Flame Retardancy

HR – High Frequency

LOI–Limiting oxygen index

MH – Magnesium Hydroxide

O_2 – Oxygen

octaBDE – octabromodiphenyl ether

PA – Polyamide

PAD – Polyamide

PAN – Polyacrylonitrile

PBDEs – Polybrominated Diphenyl ether

PC – Polycarbonate

(PCBs) – Polychlorinated Biphenyls

PE – Polyethylene

PES – Polyester

PET – Polyethylenetelephthalate

PU – Polyurethanes

PUT – Purified terephthalic acid

PVC – Polyvinylchloride

Sb – Antimony

Tg – Glass Temperature

Tm – Melting Temperature

TPU – Thermoplastic Urethanes Elastomer

WBPU – Waterborne Polyurethane

WVP – Water Vapour Permeability

Definitions

AC (Acrylic). Rigid plastic with a high degree of transparency. Resistant to inorganic acids and alkalis but is attacked by a wide range of organic solvents. Good mechanical strength and dimensional stability, along with high tensile and flexural strength and good surface hardness for scratch resistance.

Additive. Compound added after the polymer has been synthesized but before or during its conversion to final form (e.g., fiber, plastic); not covalently bound to polymer substrate.

Afterglow. Flameless or smouldering combustion.
Fire Resistance. Capacity of a material or structure to withstand fire without losing its functional properties.

Flame Resistance. Property in a material of exhibiting reduced flammability.

Flame Retardancy. Resistant to catching fire. Helps delay or prevent combustion. Fire retardants used are chemical retardants, including fire-fighting foams and fire-retardant gels.

Flammability. A measure of a materials propensity to burn or, conversely, its resistance to ignition.

Heat release rate. The thermal energy released per unit time by a material during combustion under specified test conditions.

(Limiting) oxygen index. Minimum percent oxygen in the environment which sustains burning under specified test conditions.

Vertical, horizontal, 45" [test]. Orientation of the test specimen during flammability under specified conditions.

Self-extinguishing. Does not continue to burn under the specified test conditions after the source of ignition is removed (under specified test conditions).

PU (Polyurethanes). Very variable compositions; properties range from hard, inflexible plastic to soft, elastic coatings. Plasticizers not required. Some grades have good resistance to fuels and oils. Excellent strength and resistance to tearing and abrasion. Thermoplastic grades available. Moderate to high cost.

Pyrolysis. Irreversible chemical decomposition caused by heat usually without oxidation. Pyrolysis of polymers can produce shorter-chain polymers (lower molecular weight) or the original monomer.

Rate of heat release. Amount of heat released per unit time by specimen burning under specified test conditions.

Toxicity. Harmful effect on a biological system caused by a chemicals or physical agents.

1. Introduction

Flame retardants are materials that inhibit or resist the spread of fire. There are other materials that are resistant to small open flame and that only depends to their physical and chemical structure. The use of flame retardant additives makes other material to be resistant to flame. These additives maybe applied on the material by means of coating spraying and other many ways. [1]

Thermoplastics are materials that are considered to be highly flammable so flame retardant addictives are applied on them to reduce the risk of fire. Use of flame retardants plays a major role in fire safety. Fire releases both hot gases and radiated heat. When these raise the temperature in a room to around 600°C, then all non flame retarded flammable materials present (textiles, carpets, furnishings, plastics will spontaneously ignite. Type of flame retardant used depends on the material and also a degree of fire is considered. There are many different flame retardants, and work in a number of different ways. Some flame retardants are effective on their own; other products are used mainly to increase the effect of other types of flame retardant. Minerals such as asbestos, compounds such as aluminium hydroxide, magnesium hydroxide, hydromagnesite, antimony trioxide, various hydrates, red phosphorus, and boron compounds, mostly borates resist the spread of fire.

Many of chemicals used are harmful because of their linkage to liver, thyroid, reproductive/developmental, and neurological effects and so they are burned from being used and some many chemicals also. There are studies that have been carried out and show that modern flame retardants, when appropriately applied, can be used in consumer products without significant risk to human health or the environment. [2]

There are various organizations throughout the world creating fire standard including both national and international organizations. In polymer combustibility, there is Ignitability tests, Flame spread tests, Oxygen Index, Heat release tests, Smoke tests. [3]

2. Theoretical part

2.1. Flame Retardancy

2.1.1. Mechanism of flame retardant

It is necessary that coated fabrics should be flame retardant, in some various applications. There are different test for measuring flammability of a fabric. The fibre's temperature increases until the pyrolysis temperature, TP, is reached when heat is applied. Then fibre undergoes irreversible chemical changes, producing non-flammable gases (carbon dioxide, water vapour and the higher oxides of nitrogen and sulfur), carbonaceous char, tars (liquid condensates) and flammable gases (carbon monoxide, hydrogen and many oxidisable organic molecules). The tars also pyrolyse as temperature continues to rise producing more non-flammable gases, char and flammable gases. Eventually, the combustion temperature, TC, is achieved. The speed of heat released determined the burning behavior of textile than the amount of heat. [4]

There are several approaches to attempt to disrupt this cycle: One method is to provide a heat sink on or in the fibre by use of materials that thermally decompose through strongly endothermic reactions. If enough heat can be absorbed by these reactions, the pyrolysis temperature of the fibre is not reached and no combustion takes place. Examples of this method are the use of aluminum hydroxide or 'alumina trihydrate' and calcium carbonate as fillers in polymers and coatings. Another approach is application of material that forms an insulating layer around the fibre at temperatures below the fibre pyrolysis temperature. Boric acid and its hydrated salts function in this capacity.

When heated, these low melting compounds release water vapour and produce a foamed glassy surface on the fibre, insulating the fibre from the applied heat and oxygen. A third way to achieve flame retardancy is to influence the pyrolysis reaction to produce less flammable volatiles and more residual char.

This condensed phase mechanism can be seen in the action of phosphorous-containing flame retardants which, after having produced phosphoric acid through thermal decomposition, crosslink with hydroxyl-containing polymers thereby altering the pyrolysis to yield less flammable by-products.[5]

2.1.2 Combustion process

The stages of combustion are ignition, growth, propagation and finally decay, but every fire in real life situations is unique because the circumstances and conditions are never exactly the same. The way fabrics burn depends upon a variety of factors and combinations of factors, including fabric stiffness, drape, contact with or proximity to other materials, supply of air, draughts, etc. Smoke results from the incomplete burning of materials and is a dispersion of solid or sometimes liquid particles, together with gases, some formed by the combustion process. [6]

Fire is a hazard, not only because of the danger of contact with flames, but also because of suffocation by toxic fumes, injury from heat levels and heat stress, plus all the dangers associated with panic and the inability to escape because routes are obscured by dense smoke. Individual test methods have been devised to take all these factors into consideration, some of them after lessons learnt in actual disasters. [7]

The flammability behavior of a material can be described by the following number of factors;

- ignition or how readily a material ignites.
- how rapidly fire spreads across a surface.
- how much heat is released and how quickly or heat release rate (HRR)
- how rapidly fire penetrates a wall or barrier.
- how rapidly/easily the flame chemistry leads to extinction.
- smoke production; the amount, evolution rate and composition of smoke during the stages of a fire. [5]
- toxic gas production; the amount, evolution rate and composition of gases released

during the stages of a fire.

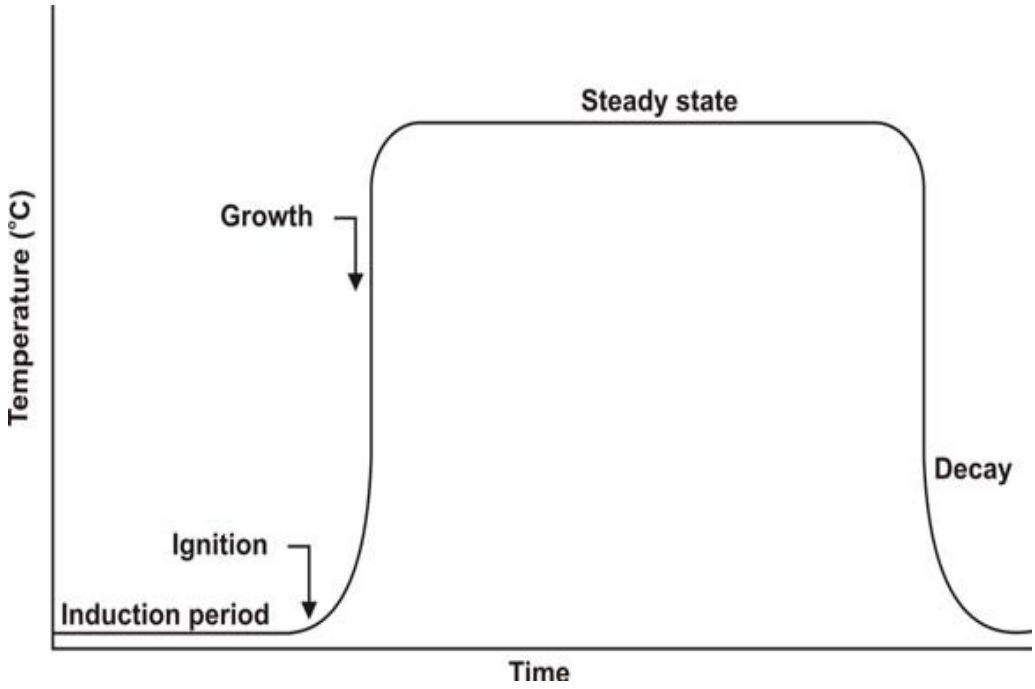


Fig 2.1 Stages of combustion [8]

The three main constituents of the fire triangle are fuel, energy (heat), and oxidant (oxygen). [9] The diffusion flame is a result of oxygen diffusing into the gaseous fuel. Smouldering combustion is the oxidation of a solid without a flame, which usually results in an increase in temperature and/or the production of smoke. Spontaneous combustion or ignition is the process by which oxygen combines slowly with the fuel, usually at its surface, with the slow evolution of heat energy. [8]

2.1.3 Char

Char is a solid material that remains after light gases have been driven-out or released from carbonaceous material during the initial stage of combustion. In the development of char the fire stops. The char has porous structure that prevents flame vapors from changing into flame. Insulation layer is formed keeping the polymer from melting below its decomposition temperature. Char can be a good thermal insulator if density is low and porosity is high, also slowing thermal decomposition process. Char provides protection during combustion and that also depends on chemical and physical structure although char from polymer combustion do not have this property. [10]

There is ideal and non ideal structure of char. Ideal char is a complete structure of the cells is closed contains pockets of gas. The polymer melt is thickened and solidifies producing honey-combed structure preventing volatile liquids into the flame. Non ideal char structure is the opposite of ideal char structure because it does not have closed cells. It has channels through which polymer melt can escape. More important to both of these effects the importance is the movement of liquid. [11]

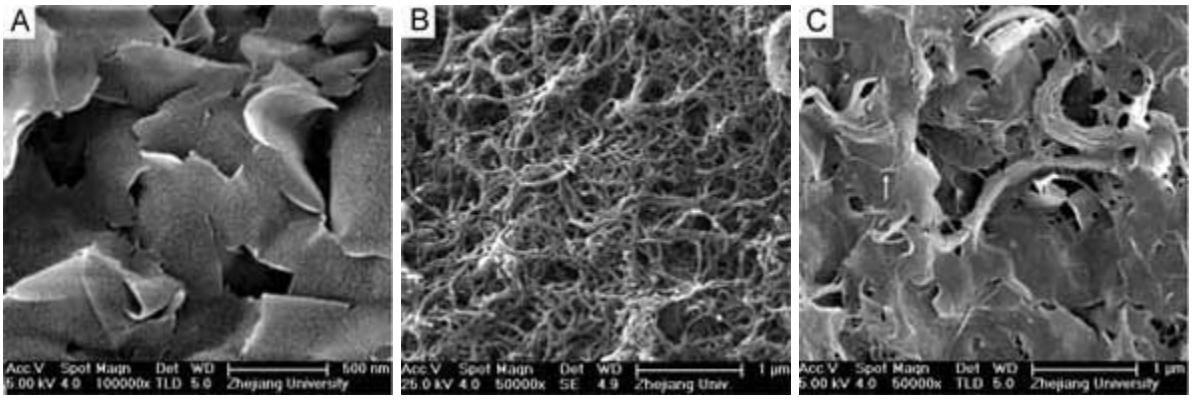


Fig. 2.2 Scanning electron microscopic images of the chars of ABS nano-composites after cone calorimeter tests: ABC

2.1.3.1. Intumescent materials

When the material is set on fire where chemical reactions result in melting, tiny bubbles cause the material to swell causing and then solidify. An intumescent coating protects the underlying substrate from fire through two mechanisms: heat is absorbed by the endothermic chemical reactions that produce the bubbles, and the low thermal conductivity of the bubbles provides an insulating layer. Char acts as a physical barrier, protecting the polymer against heat transmission, diffusion of oxygen toward the surface and diffusion of combustible degradation products of the polymer away from the surface, outward toward the flame. The chemical mechanism causes a plateau in the plot of substrate temperature vs. time, and the decreased effective thermal conductivity slows the temperature increase with time for the final char layer. [11]

2.1.4 Use of different textiles

2.1.4.1. Cotton fibres

Cotton is the most important fibre used in the world. Cotton is essentially cellulose like any plant fibers. Many of everyday textiles are made of cotton. They are capable of infinite variety of weave and coloring. Cotton fabrics were made by Ancient Egyptians and by the earliest of Chinese civilizations. [12]

Cotton, as a natural cellulosic fiber, has a lot of characteristics, such as;

- Comfortable Soft hand
- Good absorbency
- Color retention
- Prints well
- Machine-washable
- Dry-cleanable

- Good strength
- Drapes well
- Easy to handle and sew

Each cotton fiber is composed of concentric layers. The cuticle layer on the fiber itself is separable from the fiber and consists of wax and pectin materials. The primary wall, the most peripheral layer of the fiber, is composed of cellulosic crystalline fibrils. The secondary wall of the fiber consists of three distinct layers. All three layers of the secondary wall include closely packed parallel fibrils with spiral winding of 25-35^{o} and represent the majority of cellulose within the fiber. The innermost part of cotton fiber- the lumen and it is the hollow canal that runs the length of the fiber. It is filled with living protoplast during the growth period. After the fiber matures and the boll opens, the protoplast dries up, and the lumen naturally collapses, leaving a central void, or pore space, in each fiber. [13]

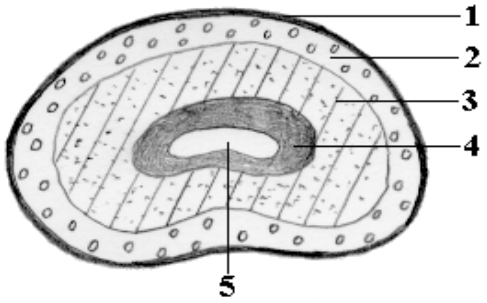


Fig. 2.3 Bean shaped cross section through a cotton fiber

1 - Wax layer
2 - Primary wall
3 - Secondary wall, up to approximately 94% cellulose
4 - Tertiary wall
5 - Lumen (cavity), air-filled

2.1.4.2 Chemical composition of cotton fibres

Average cotton composition: 86 – 96%cellulose, 2-3%peptides, 0, 4 - 1, 2%pectin, 0, 4 –

0,8%fats and waxes, 1 – 1, 8%minerals, 6 – 8, 5%water (humidity), pigments. Basic properties of cotton: Between –OH groups are Hydrogen bond = intramolecular (between OH groups of one molecule) and intermolecular (between to molecules) forces. Cellulosic fibers are damaged by oxidative chemicals, acids and under alkali solution (only at high temperature and together with air oxygen). All these chemicals degradate cellulose polymer. The function groups in cellulose are changed and the polymerization degree is reduced. Mechanical effects: reduction of mechanical properties (low strength) and the fibers are more sensitive to future damage. Chemical damage changes of polymer chemical properties. Change of chemical groups, change of polymerization degree. Oxycellulose changes aldehyde and carboxyl groups.

Hydrocellulose changes aldehyde groups. Mechanical damage changes of fiber geometry. Change of ends of fibers, appearance of fibers. In solution of NaOH under Microscopic observation: undamaged or mechanical damaged cotton swell on the ends of fibers, low chemical damaged cotton swell along the fiber and high chemical damaged cotton fiber decomposition. [14]

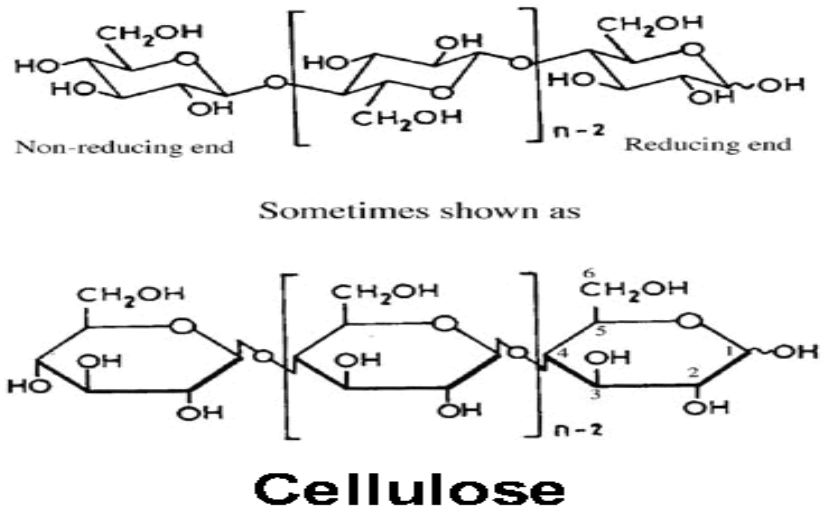


Fig. 2.4 Carbon „6“– primary hydroxyl group; Carbon „2“and „3“– secondary hydroxyl group

At temperatures > 25°C, cotton dries out, becomes hard and brittle and losses elasticity. Light causes the same deterioration. The optimum temperature for mold development is 25 - 35°C. Cotton is subject to self-heating/spontaneous combustion. The auto ignition temperature of oily cotton is 120°C. At temperatures < 0°C there is no risk of wet bales rotting, since this

process stops at low temperatures. In some cases, damaged cotton has been placed in intermediate cold storage, so preventing rot. Every hold should be equipped with means for measuring temperature. Measurements must be performed and recorded daily. Qualitative analysis of cotton: It burns and may flare up when lit. No melted bead is left by it. After burning, it continues to glow. It gives out smell like that of a burning paper. The smoke is gray or white. The ash is fine, soft that can be easily crumbled. [12]

2.1.4.3 Flame retardants for cotton fibres

Untreated natural fibers such as cotton, linen and silk burn more readily than wool, this is more difficult to ignite and burns with a low flame velocity. Cotton also has a high burning rate but this can be alleviated by the application of flame-retardant chemical additives. One important thermal degradation mechanism of cellulose fibres (cotton, rayon, linen, etc.) is the formation of the small depolymerisation product levoglucosan (fig.2.6). Levoglucosan and its volatile pyrolysis products are extremely flammable materials and are the main contributors to cellulose combustion. Compounds that are able to hinder levoglucosan formation are expected to function as flame retardants for cellulose. The cross-linking and the single type of esterification of cellulose polymer chains by phosphoric acid reduces levoglucosan generation, catalyses dehydration and carbonization, and thus functions as an effective flame retardant mechanism. Solubility and durability are important issues, the ideal non-durable finish should be able to penetrate the fibre and minimize surface deposits. Then; the release of Lewis acidic properties should not occur significantly below 150 °C if the treated textile is to resist normal drying and curing temperatures. Finishes like borax ($Na_2B_4O7.10H_2O$) and boric acid (H_3BO_3) in a 7:3% (w/w) ratio at add-ons of 10–15% (w/w) have been used for many years [32], this finish does start to decompose and release acid above 130 °C and has poor afterglow retardancy.

Afterglow must be prevented because it may lead to the slow burning of the fibers, this can revert back to flaming combustion if there is sufficient air present. [13]

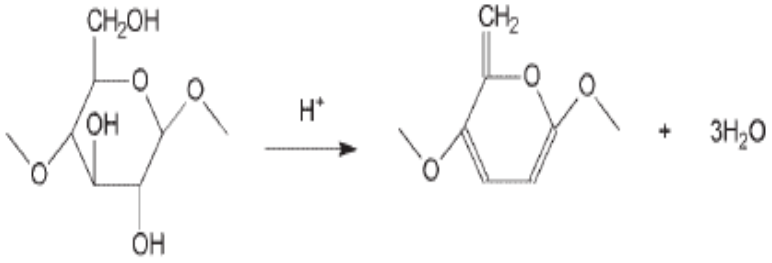


Fig. 2.5. Dehydration of cellulose by strong acids.

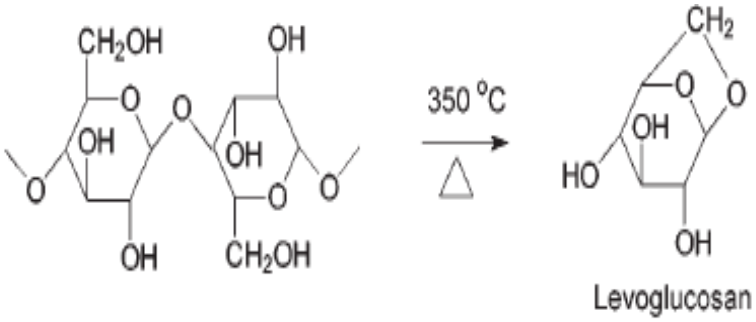


Fig.2.6. Thermal degradation of cellulose

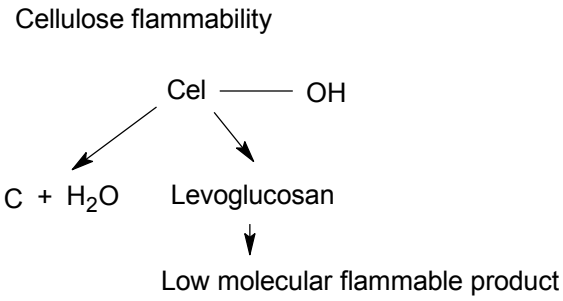


Fig.2.7 Up to 200 °C depolymerization, 200-300 °C pyrolysis, from 350 °C burning, od 400 °C selfignition

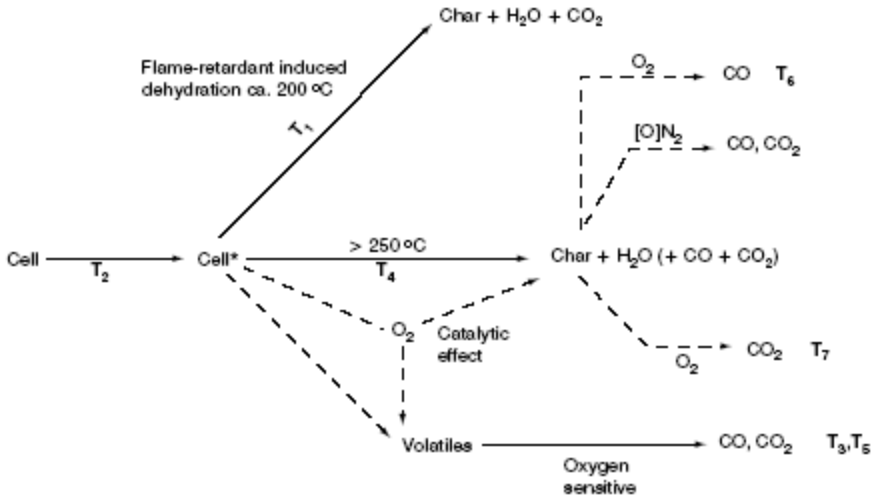


Fig. 2.8 Schematic modified mechanism for cellulose pyrolysis

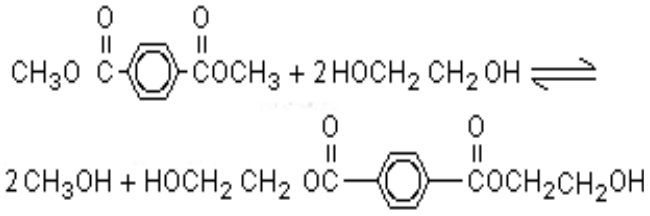
2.1.4.4. Polyester fibres

Polyester is a category of polymers which contain the ester functional group in their main chain. Natural polyesters and a few synthetic ones are biodegradable, but most synthetic polyesters are not. Depending on the chemical structure polyester can be a thermoplastic or thermoset, however the most common polyesters are thermoplastics. Polyester has several advantages over cotton. It absorbs oil, but it does not absorb moisture; this quality makes polyester the perfect fabric for the application of water-, soil-, and fire-resistant finishes. Its low absorbency also makes it naturally resistant to stains. Polyester clothing can be preshrunk in the finishing process, and thereafter the fabric resists shrinking and will not stretch out of shape. The fabric is easily dyeable, and not damaged by mildew. [14]

The qualitative analysis of polyester: It is a polymer produced from coal, air, water and petroleum products. It burns quickly and shrinks away from flame, may also flare up. It leaves hard, dark, and round beads. After the flame, it burns slowly and is not always self-extinguishing. It has a slightly sweet chemical odor. It leaves no ash but its black smoke and fume are hazardous. Textured polyester fibers are an effective, nonallergenic insulator, so the material is used for filling pillows, quilting, outerwear, and sleeping bags. Polyester polymer is produced

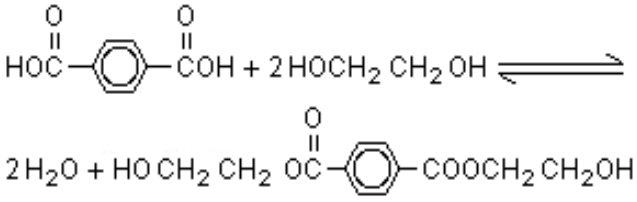
commercially in a two step polymerization process, i.e., monomer formation by ester interchange of dimethyl terephtalate with glycol or esterification of terephthalic acid with glycol followed by polycondensation by removing excess glycol: [15]

STEP 1: Ester interchange:



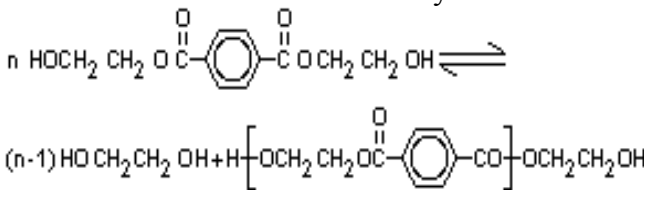
(1)

Esterification



(2)

STEP 2: Polycondensation



(3)

2.1.4.5 Chemical composition of polyester fibres

The most common polyester for fiber purposes is poly (ethylene terephthalate). A manufactured fiber in which the fiber forming substance is any long-chain synthetic polymer composed of at least 85% by weight of an ester of a substituted aromatic carboxylic acid. [16]

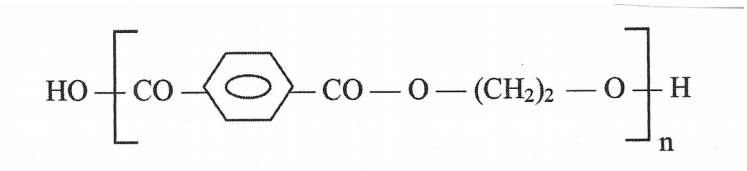


Fig. 2.9 Basic structural unit of polyethyleneterephtalate

Effect of alkali's: Polyester fibres have good resistance to weak alkali's, high temperatures. It exhibits only moderate resistance to strong alkalis' at room temperature and is degraded at elevated temperatures. Effect of acids: Weak acids, even at the boiling point, have no effect on polyester fibres unless the fibres are exposed for several days. Polyester fibres have good resistance to strong acids at room temperature.

Exposure to boiling hydrochloric acid destroys the fibres and 96% sulfuric acid and causes disintegration of the fibres. Effect of solvents: Polyester fibres are generally resistant to organic solvents.

Chemicals used in cleaning and stain removal do not damage it, but hot m-cresol destroys the fibres, and certain mixtures of phenol with trichloromethane dissolve polyester fibres. Oxidizing agents and bleachers do not damage polyester fibres. Miscellaneous properties: Polyester fibres exhibit good resistance to sunlight, and it also resists abrasion very well. Soaps, synthetic detergents, and other laundry aids do not damage it. One of the most serious faults with polyester is its oleophilic quality. It absorbs oily materials easily and holds the oil tenaciously. The moisture regain of polyester is low, ranges between 0.2 to 0.8 percent. Heat effect: The melting point of polyester ranges from 250 to 300°C. Polyester fibres shrink from flame and

melt, leaving a hard black residue. The fabric burns with a strong, pungent odor. Heat setting of polyester fibres, not only stabilizes size and shape, but also enhances wrinkle resistance of the fibers. [17]

It made of purified terephthalic acid (PTA) or its dimethyl ester dimethyl terephthalate (DMT) and monoethylene glycol (MEG). Poly (ethylene terephthalate or simply PET) is the most common polyester used for fiber purposes. [16]

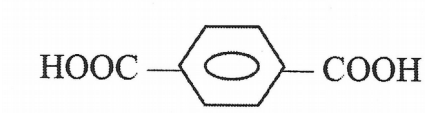


Fig. 2.10 Terephtalic acid

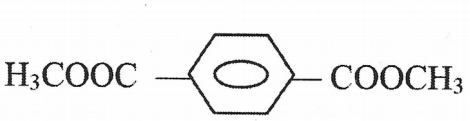


Fig. 2.11 Dimethylterephtalate

An increase in molecular weight further increases tensile strength, modulus and extensibility. Shrinkage of the fibres also varies with the mode of treatment. If relaxation of stress and strain in the oriented fibre occurs, shrinkage decreases but the initial modulus may be also reduced. Yarns maintained at a fixed length and constant tension during heat setting is less affected. Synthetic materials have higher temperature glass and for polyester is 75 Tg (°C).

In textile polyester fibres have taken the major position although they have many drawbacks e.g,

(a) Low moisture regains (0.4%),

(b) The fibres has a tendency to accumulate static electricity,

(c) The cloth made up of polyester fibres picks up more soil during wear and it also difficult to clean during washing,

(d) The polyester garments from pills and thus, the appearance of a garment is spoiled,

(e) The polyester fibre is flammable with respect to changes in modulus, and reduced shrink-age values are still obtained. [17]

2.1.4.6. Flame retardant for polyester fibres

Polyester tends to be slow to ignite but once ignited, severe melting and dripping occurs. Since the fabric melts away from the flame, some polyester fabric constructions can actually pass vertical flame tests without any flame-retardant treatment. The waiving of melt– drip specifications for children's sleepwear has allowed untreated polyester garments to be sold into that market. One of the most useful flame-retardant finishes for polyester was bromine containing phosphate ester, trisdibromopropylphosphate and was eventually removed from the marketplace by legislation.

The current flame retardant used for polyester is the mixture of cyclic phosphate/ phosphonates used in a pad–dry–heat set process (fig 2.12) .Heat set conditions of 190 – 210ºC for 0.5 – 2 min are adequate. This product when applied at ~ 3–4 % add-on can provide durable flame retardancy to a wide variety of polyester textiles.

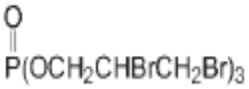


Fig 2.12 Tris (2, 3-dibromopropyl) phosphate

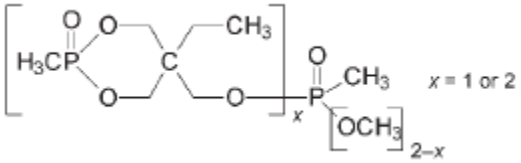


Fig 2.13 Cyclic phosphate / phosphonate flame retardant.

Polyester

T_g= 83°C T_m = 266°C Tp = 451°C

Pyrolysis products – CO_2 + CO + acetaldehyde + terefthalic acid and benzen acid [18]

2.1.4.7. Blends of polyester/cotton fibres

Cotton - poly fabric is a made combining strands of cotton and polyester. It can be 65% cotton and 35% polyester. This blend is usually quite comfortable by combining the natural effects of cotton for softness and moisture absorption with the no-iron crispness of polyester. The behavior of polyester/cotton blended system is different to those obtained with the separated system. The chemical composition of gaseous pyrolysate is different with the blended material materials than found with the separated systems. The magnitude of pressure rise on ignition of the pyrolysate/air mixtures may be taken as a useful indicator of the energy being released during the combustion process and is therefore capable of providing information on the potential heat feedback during the burning process.

In the case of separated systems, the energy released during ignition of the pyrolysate closely resembles that of cotton alone, a fact to be expected based upon the data obtained which indicate that the cotton is the source of the pyrolysate. But polyester/cotton blends, the energy release as measured by the maximum pressure rise on ignition lie above the line connecting the values for the individual components with two of the blends having values greater than that for polyester alone. Polyester/cotton fabric absorbed higher amounts by mass methomyl than nylon or PVC fabrics. Each fiber absorbs different quantity of water according its chemical composition. The quantity of absorbed water is connected with their humidity. Higher air humidity is equal to high contain of water in fibers. The fibers change its weight according the relative humidity of air. [19]

2.1.4.8 Flame-retarding fibre blends

Fibre blends, especially blends of natural fibres with synthetic fibres, usually exhibit a

flammability that is worse than that of either component alone. Natural fibres develop a great deal of char during pyrolysis, whereas synthetic fibres often melt and drip when heated. Burning of fibres blends consisting thermoplastic and nonthermoplastic fibresis a grid effect; the melt of thermoplastics absorbs into nonthermoplastic fibres and the substrate becomes more flammable. This combination of thermal properties in a fabric made from a fibre blend results in a situation where the melted synthetic material is held in the contact with the heat source by the charred natural fibre. Cotton fibre char acts as a candle wick for the molten synthetic material, allowing it to burn readily. [20]

The current rules for the simple flame-retarding of blends are either to apply flame retardant only to the majority fibre present or apply halogen-based back-coatings, which are effective on all fibres because of their common flame chemistries in the vapour phase. In the case of durable, phosphorus-containing cellulose flame-retardants, they are generally only effective on cellulose-rich blends with polyester.

However, the use of a cotton-rich blend here is particularly advantageous because the lower polyester content confers a generally lower thermoplastic character to the fabric with a smaller tendency to produce an adhesive molten surface layer when exposed to a flame. This can be demonstrated by the LOI values of cotton18, polyester 20 and a 50/50 blend of both indicate a higher flammability.[21]

Table 2.1 Thermal transitions of the more commonly used fibres

Fibre)	*T*g (softens) (°C)	*T*m (melts) (°C)	*T*p (pyrolysis) (°C)	*T*c (ignition) (°C)	Limiting oxygen indexLOI (%)
Wool	-	-	245	570-600	25
Cotton	-	-	350	350	18.5
Viscose	-	-	350	420	18.9

Nylon 6	50	215	431	450	20-21.5
Nylon 6.6	50	265	403	540	20-21.5
Polyester	80-90	255	420-447	480	20-21
Acrylic	100	>220	290 (with decomposition)	>250	18.2
Polypropylene	–20	165	470	550	18.6
Modacrylic	<80	>240	273	690	29–30
PVC	<80	>180	>180	450	37-39
Meta-aramid (e.g. Nomex)a	275	375	410	>500	29–30
Para-aramid (e.g. Kevlar)a	340	560	>590	>550	29

a Fibers manufactured by Du Pont

2.2 Flame retardant system

2.2.1 Evaluation of flame retardant treatments

The flammability of textiles is influenced by many factors, including the fibre type, the fabric weight and construction, the method of ignition, the extent of heat and material exchange, and the presence or absence of flame retardants. [13]

Prevention of flame retardants or ignition of materials saves many lives. There are concerns related to chemical release into the environment, degradation products or potential health effects. (PCB)polychlorinated biphenyl were banned in 1977 because its harmful and the EU has banned several types of brominated flame retardants as of 2008, following evidence beginning in 1998 that the chemicals were accumulating in human breast milk. Currently some US states and various countries are investigating PBDEs as well; of the major ones only decaBDE remains on the North American market. Since halogenated flame retardants have been in the focus of public scrutiny, flame retardants based on other chemistries like phosphorus and nitrogen have been developed and need to prove their environmental benefits. [7]

2.2.1.1 Phosphorus flame retardant

There is a range of phosphonates which are used mainly in applications such as rigid polyurethane foam. These are products with water like viscosities. The products are nonreactive. Among non-toxic flame retardant coatings, phosphorus- containing coatings have replacement of halogen containing coatings. The most common are dimethyl methyl phosphonate, diethyl ethyl phosphonate, dimethyl propyl phosphonate and diethyl N, N – bis (2-hydroxyethyl) amino methyl phosphonate. These products are used exclusively in rigid polyurethane foam. It is reactive and becomes part of the polyurethane polymer during processing. Phosphorus-based flame retardants can be organic, inorganic, or elemental. They can be active in the vapor phase or in the condensed phase, or sometimes in both phases.

Phosphine oxides and phosphate esters are thought to act in the vapor phase through the formation of PO* radicals, which terminates the highly active flame propagating radicals (OH* and H*). [3]

The condensed phase mechanism arises as a consequence thermal generation of

phosphoric acids from the flame retardant, e.g. phosphoric acid or polyphosphoric acid. These acids act as dehydrating agents, altering the thermal degradation of the polymer, and promoting the formation of char.

Generic molecular structures in which phosphorus flame retardants are based on:

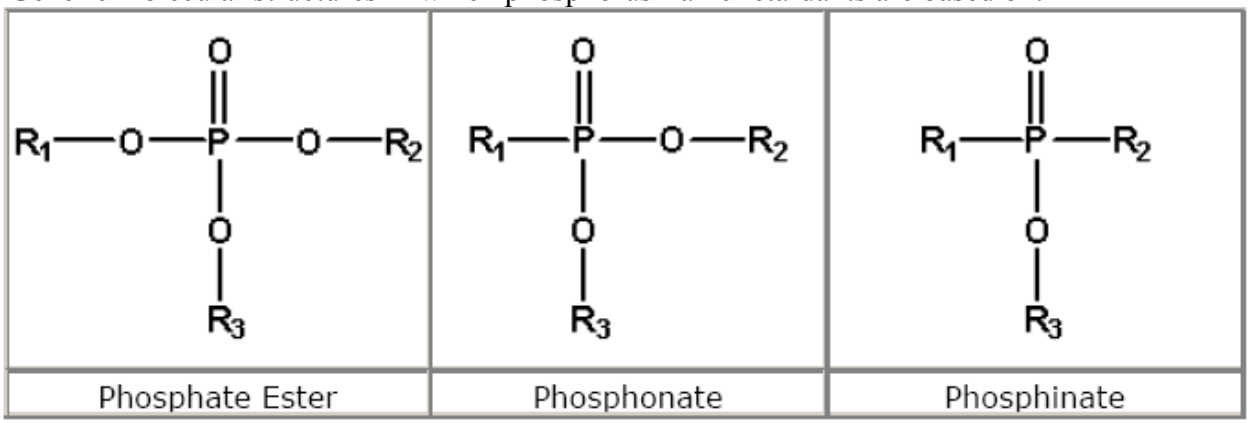


Fig. 2.14 R1, R2, R3 are organic substituent's, they can be different or the same

Phosphorus compounds form a protective layer in a solid or gaseous protective layer which excludes the oxygen necessary for the combustion process. Also reaction in solid phase is by forming carbonaceous layer on the polymer surface. [22]

2.2.1.2 Aluminium trihydrate and magnesium hydroxide

Mostly used fire retardant for polymers are alumina trihydrate, ATH, ($Al_2O_3 \cdot 3H_2O$), magnesium hydroxide, MH, ($Mg(OH)_2$) and Zinc borate. One of the advantages of aluminium trihydrate is low cost. It is applied in thermosetting resins and because of that it's limited, especially to polymers below 200°C. Magnesium hydroxide is doing the opposite being stable to polymers above 300°C. They both decompose endothermically and consume a large amount of heat, while also liberating water, which can dilute any volatiles and thus decrease the possibility of fire. [23]

$$2Al(OH)_3 \longrightarrow Al_2O_3 + 3H_2O \quad (4)$$

Aluminium trihydrate decompose at 300°C while magnesium hydroxide, decomposes at

400°C. Both aluminium trihydrate and magnesium hydroxide are in low smoke, halogen free wire and cable applications. With polypropylene, 60% loading of MH gives an oxygen index of 26, while with polyamide-6; the same loading gives an oxygen index of almost 70.

Magnesium Hydroxide produces more char than aluminium trihydrate resulting in increased effectiveness and less smoke and dilutes the amount of fuel available to sustain combustion. It during combustion generates highly reflective magnesium oxide coating which deflects the flame's heat away from the polymer. [24]

$$Mg(OH)_2 \longrightarrow MgO + H_2O \quad (5)$$

2.2.1.3 Zinc borate

Zinc Borate can be used as a fire retardant in PVC, polyolefins, elastomers, polyamides, epoxy resins. Zinc borate-boron containing compounds act through the endothermic, stepwise release of water and by the formation of glassy coating protecting the substrate. In halogen–free system zinc borate is used is normally used in conjunction with aluminium trihydrate and also can be used as multi-functional synergistic additives with other flame retardant additives in polymers to improve the flame retardant performance, reduce smoke evolution and adjust the balance of flame retardant properties versus mechanical, electrical and other properties. Other application areas are in nylon engineering plastics for electronic and electrical components, halogen free flame retardant systems based on alumina trihydrate and magnesium hydroxide and intumescent coatings and paints. Zinc borates display low acute toxicity. [23]

2.2.1.4 Flame retardants based on nitrogen compound

Nitrogen compounds are a small but rapidly growing group of flame retardants (FR) which are in the focus environmentally friendly flame retardants. Their main applications are melamine for polyurethane flexible foams, melamine cyanurate in nylons, melamine phosphates in polyolefines, melamine and melamine phosphates. Their main common advantages are their

low toxicity, their solid state and, in case of fire, the absence of dioxin and halogen acids as well as their low evolution of smoke. Flame retarded materials based on nitrogen compounds are suitable for recycling as the nitrogen flame retardants have high decomposition temperatures. [24]

2.3 Applications used for flame retardant textiles

2.3.1 Coating methods

Coating material consists of a number of large individual substances; polymers cross linking agents, rheology flow and structuring additives, reaction accelerators or inhibitors, light protection agents, pigments and effect agents. The additives mainly support the application and film forming processes while the role of pigment and effect substances it meet the visual requirements of coatings specifically, the technological requirements in automotive coatings made from paints such as scratch resistant flexibility or resistant to yellowing must be achieved by means of polymer system or polymers and cross-linking systems used in the coating materials. Polymers and cross-linking agents contain complimentary functional group that reacts with each other under relevant specified reaction condition to form huge skeleton of the cured coatings. Three dimensional networks have a very significant on property profile of coating. [25]

Now it is clear not only parameters such as closeness of the mesh, number of points of the linkage but also type of mesh material used must of necessity have a decisive effect on the property profile. In automotive paints, the very high scratch resistance is currently targeted for example is the function of both the glass transition temperature and the cross-link density. A layer of a polymer coated on the substrate imparts new characteristics to the base fabric. The resultant coated fabric may have functional properties such as resistance to soiling or penetration of a fluid, or it may have entirely different aesthetic appeal, such as finished leather. There are various coating methods by which to apply polymer to textiles. They can be classified on the

basis of equipment used, method of melting and foam of coating materials. These methods are as follows: [26]

Table 2.2 Classification of basic equipment used for coating

COATING METHOD	Coating thickness (mils)	Viscosity (centipoise)	Speed Range (fpm)	Width (inches)
Knife Over Roll (Kor)	1 to 30	1,000 to 30,000	0 to 300	0 to 120
Gravure	2 to 5	50 to 10,000	10 to 1200	0 to 80
Offser Gravure	1 to 5	100 to 10,000	10 to 1000	0 to 80
Reverse Roll	1 to 20	2,000 to 30,000	30 to 1200	0 to 100
Multi- Roll	0.2 to 2	100 to 25,000	100 to 1200	0 to 80
Slot Die	0.5 to 40	10 to 1,000,000	0 to 12000	0 to 80

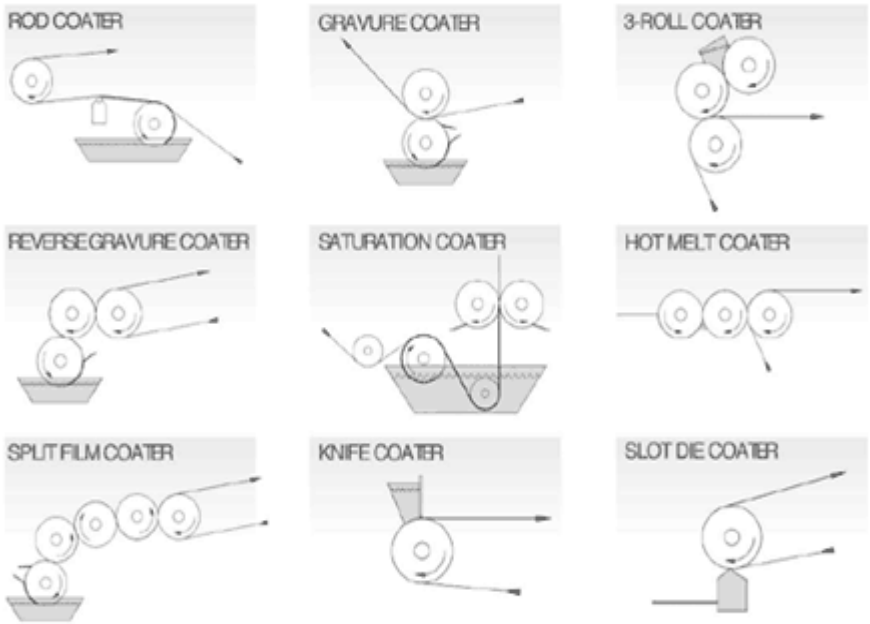


Fig. 2.15 Different kinds of coaters

1. Fluid coating (coating material is in the form of paste, solution or lattices)

a) **Knife coaters:** wire wound bars, round bars, floating knife and so forth. To get a perfect coating result without any "spit-balls", the design of the doctor blade should be selected according to the coating method and used coating material and substrate

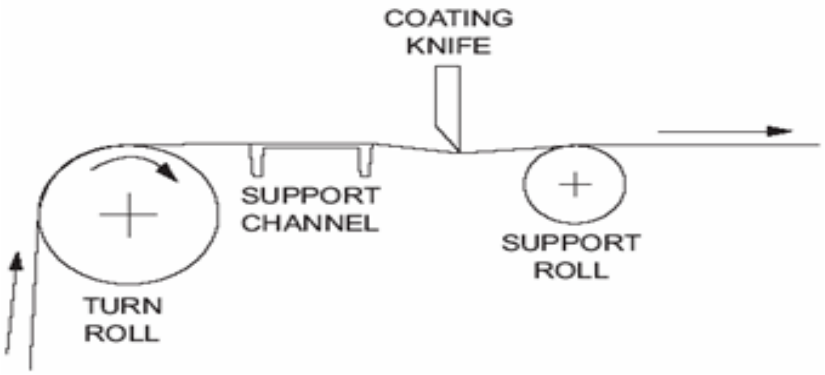


Fig. 2.16 Floating air knife

Position of the coating knife

- Higher add-on due to higher pressure on the paste in front of the knife.
- Lower add-on due to lower pressure. air-knife systems is applied on airbags, apparel, protection clothings.
- Sharp knife – coating of fine fabrics
- Flat knife – paste is partly pressed into the fabric

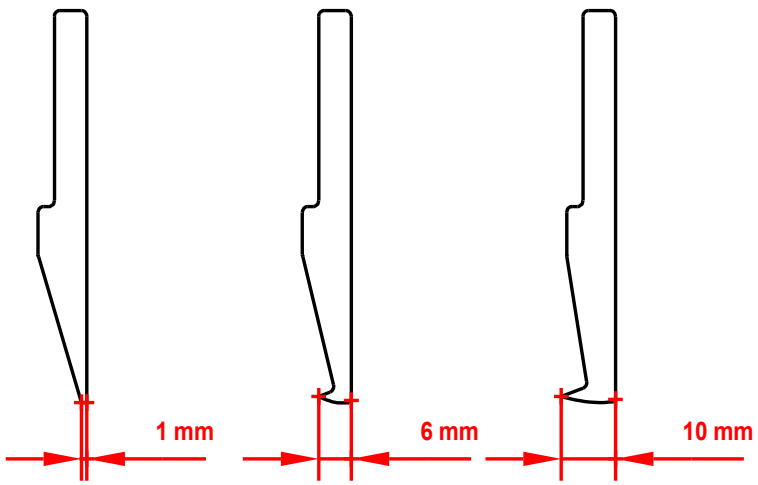


Fig. 2.17 Position of the coating knife

Low investment cost, simple application, handling and maintenance. It is suitable for paste and stable foam coating. Solid add-ons: 25 to 250 g/m² (per coat) depending on:

- Free gap
- Fiber type
- Knife type

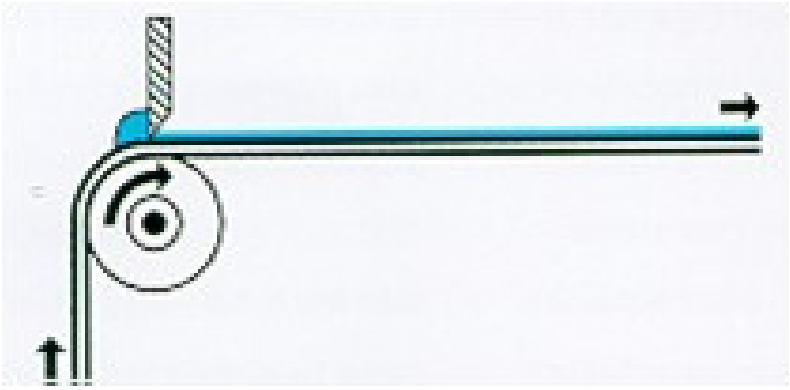


Fig. 2.18 Knife over roller

These are the post metering devices.

b) Roll coaters: Reverse roll coaters, kiss coaters, gravure coaters, dip coaters etc. The material to be coated is dipped in the fluid, and excess is removed by squeeze roll or doctor blades.

c) Impregnators: The material to be coated is dipped in the fluid, and excess is removed by squeeze roll or doctor blades.

d) Spray coater: The material is sprayed directly on the web or onto a roller for transfer.

2. Coating with dry compound (solid powder or film)

a) Melt coating: Extrusion coating, powder coating and so forth.

b) Calendaring: For thermoplastic polymers and rubber compounds, Trimmer process and Berna coater. [27]

2.3.2. The choice of coating method

The choice of coating method depends on several factors.

- Nature of the substrate
- Form of the resin and viscosity of the coating fluid
- End-product and accuracy of coating desired
- Economics of the process [26]

2.3.3 The features of the fluid coating units

In fluid coating operation, basically, the coating operation involves applying the coating fluid on to the web and then solidifying the coating. There are common features in all coating operations. The different modular sections of the coating machine are illustrated in (fig. 2.19) and described as follows: [26]

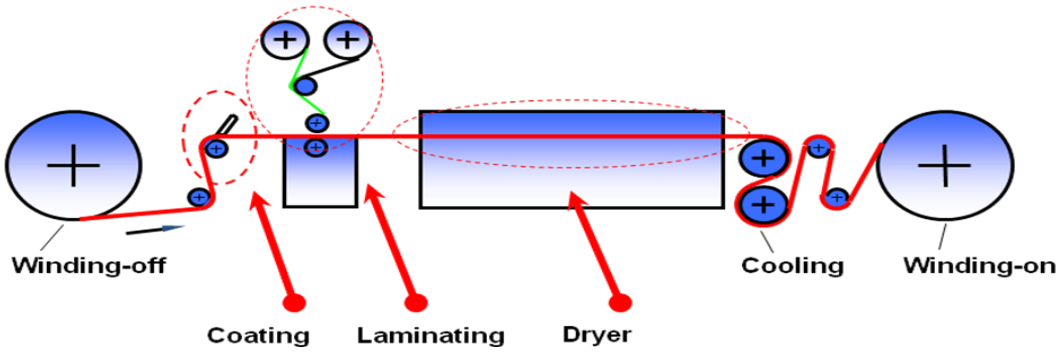


Fig. 2.19 Layout of direct coating line: winding-off (fabric let-off arrangement, coating head, laminating, dryer (drying oven), cooling, winding on.

a) Winding off. The fabric is unwind and drawn through the machine under tension.

b) Coating head. Any methods of fluid coating in this case a knife or roller can be used.

c) Laminating. Heat Roll Laminators are heated rollers that are used to melt glue extruded onto lamination film and Cold Roll Laminators use a plastic film which is coated with an adhesive and glossy backing which does not adhere to the glue.

d) Dryer. In drying oven all the solvents are evaporated and the film is solidified dried and cured. The oven can be oil or forced air heated or electrically heated. Vulcanization is carried out separately for rubber coated fabric. By IR heaters, gas-fired units, heater strips, other polymers requiring higher temperature, drying and curing can be done. Fresh air is continuously circulated through-out the oven in order to prevent volatiles from forming an explosive mixture. Drying rate is carefully controlled to prevent blister formation or cracking. Oven is divided in different zones, increasing temperature of each in order to remove the solvent without blisters and to properly control solvent evaporation.

e) Cooling. The fabric comes out of the oven hot and passes the cooling rollers to make it tack free.

f) Winding on. Fabric is winded on the roller. [26]

2.3.4. Physical properties of coated textiles

2.3.4.1. Characteristics

Coated textiles are flexible composites consisting of a textile substrate and a polymeric coating. The coating maybe on one side or both side and different polymeric coating per side. All the physical properties of coated fabric depend on the properties of substrate, the coating formulation, the coating technique, and the processing conditions during coating. A coated fabric behaves differently from both its textile part and the elastomer and can be classed a composite, and therefore properties are difficult to predict. Since the base textile has an orientation, warp or weft in a woven cloth, so then does the coated fabric. The warp yarns are aligned more parallel, where as in the weft there is an increase in the crimp. Since moisture can affect the base textile, depending on its nature (cotton, polyamide, polyester etc.) and whether it is fully encapsulated by the coating, moisture equilibrium is approached from the dry side. [28]

2.3.4.2 Tensile Strength

The strength of coated fabric is an important property, and the end use dictates the construction of the product, since the textile element mainly imparts this strength feature. For coated textiles stress is the force applied to the test specimen, and the maximum force is that recorded in extending the test piece to breaking point. The strength of a fabric depends on type of fiber, fineness, twist, and tenacity of yarns and also on the weave and yarn density. Theoretically, the tensile strength of a fabric should be the sum of the tensile strength of all the yarns added together. There is always a loss of strength due to weaving, and as a result, the theoretical strength is never achieved. [28]

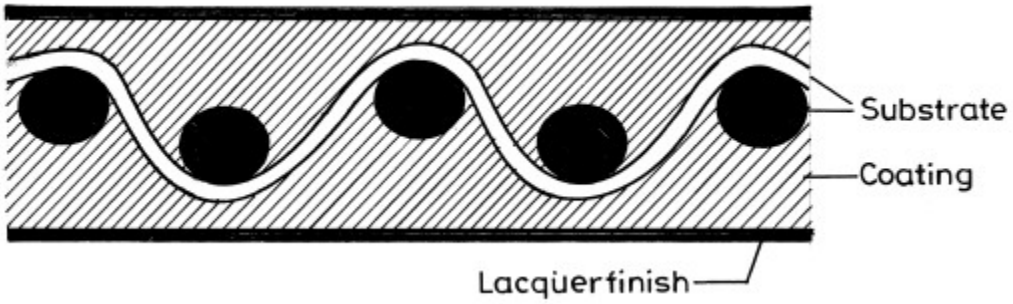


Fig. 2.20 A double-sided coated fabric.

2.3.4.3 Elongation

In automotive industry, seat covers are the most important application area of technical textiles. Higher breaking and tearing strengths and breaking elongation are specifications required for advanced seat covers. Higher breaking and tearing strengths and breaking elongation are specifications required for advanced seat covers. An increasing force is gradually applied to a textile material so that it extends and eventually breaks. Strain curve contains far more information than just the tensile strength of the material. [28]

2.4 Polymers

Polymers are substances whose molecules have high molar masses and are composed of a large number of repeating units typically connected by covalent chemical bonds. There are both naturally occurring and synthetic polymers. Among naturally occurring polymers are proteins, starches, cellulose, and latex. Synthetic polymers are produced commercially on a very large scale and have a wide range of properties and uses. The materials commonly called plastics are all synthetic polymers. Polymers are formed by chemical reactions in which a large number of molecules called monomers are joined sequentially, forming a chain. Polymers are classified by the characteristics of the reactions by which they are formed. [29]

Polymers are classified by the characteristics of the reactions by which they are formed. If all atoms in the monomers are incorporated into the polymer, the polymer is called an addition polymer. If some of the atoms of the monomers are released into small molecules, such as water, the polymer is called a condensation polymer. Most addition polymers are made from monomers containing a double bond between carbon atoms. Such monomers are called olefins, and most commercial addition polymers are polyolefins. Condensation polymers are made from monomers that have two different groups of atoms which can join together to form, for example, ester or amide links. Polymers have a range of applications. Application of polymers [30]

Agriculture and Agribusiness:

- Polymeric materials are used in and on soil to improve aeration, provide mulch, and promote plant growth and health.

Medicine

- Many biomaterials, especially heart valve replacements and blood vessels, are made of polymers like Dacron, Teflon and polyurethane.

Consumer Science

- Plastic containers of all shapes and sizes are light weight and economically less expensive than the more traditional containers. Clothing, floor coverings, garbage disposal bags, and packaging are other polymer applications.
-

Industry

- Automobile parts, windshields for fighter planes, pipes, tanks, packing materials, insulation, wood substitutes, adhesives, matrix for composites, and elastomers are all polymer applications used in the industrial market.

Sports

- Playground equipment, various balls, golf clubs, swimming pools, and protective helmets are often produced from polymers. [31]

Materials that are coated consists of substrate which has been combined with a thin, flexible film of a natural or synthetic polymeric substance. It is in a form of viscous liquid and is applied directly on the substrate. [32]

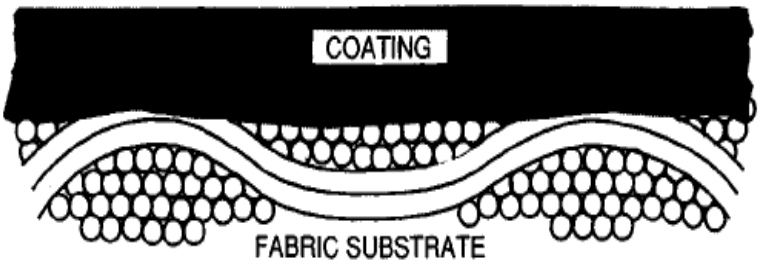


Fig. 2.21 Coated substrate combined with polymer

2.4.1 Acrylic Coating

Acrylic polymers are commonly known as acrylics. The monomers are esters of acrylic and methacrylic acid.

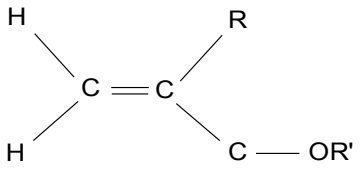


Fig. 2.22 General formula of acrylates

Above is the general formula of acrylates (R = CH_3 for methacrylates; R = H for acrylates,). Acrylic polymers tend to be soft and tacky, while the methacrylate polymers are hard and brittle. A proper adjustment of the amount of each type of monomer yields polymers of desirable hardness or flexibility. The polymerization can occur by bulk, solution, emulsion, and suspension methods. The suspension-grade polymer is used for molding powders. The emulsion and solution grades are used for coatings and adhesives. Acrylate emulsions are extensively used as thickeners and for coatings. Acrylics have exceptional resistance to UV light, heat, ozone, chemicals, water, stiffening on aging, and dry-cleaning solvents. The ester can contain functional groups such as hydroxyl, amino and amido. Acrylic acid is the common name for 2-propenoic acid: CH_2=$CHCO_2H$. Acrylic fibers such as Orlon are made by polymerizing a derivative of acrylic acid known as acrylonitrile. [33]

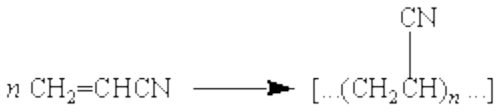


Fig. 2.23 Polyacrylonitrile

Other acrylic polymers are formed by polymerizing an ester of this acid, such as methyl acrylate.

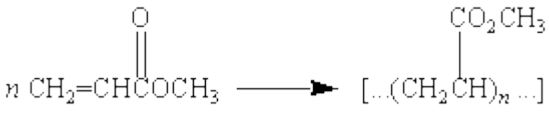


Fig2.24 Poly (methylacrylate)

The use acrylic polymers for both emulsion and solution coating are very diverse. Fundamental reason for coating is to protect the substrate and other additional reason depending to the end use area. The coating must adhere to the surface that has been coated because to protect any surface the coating must remain in position. Acrylics are available in three physical forms: solid beads, solution polymers, and emulsions. Plastics are coated to improve resistance to chemicals, solvents, ultraviolet light, and abrasion, as well as exterior durability.

Other applications for coatings include the engine enamels area, underbodies, and auto refinishing work. Acrylic polymers tend to be soft and tacky and methacrylate polymers are hard and brittle. Proper adjusted of the amount of each type of monomer yields polymers of desirable hardness or flexibility. They have exceptional resistance to UV light, heat, ozone, chemicals, water and stiffening on aging and dry-cleaning solvents. They are used as back coating materials in automotive upholstery fabric. [33]

2.4.1.1 Application and uses of acrylics

Acrylics are esters of acrylic acids, meaning they are the products formed by the reaction of an acrylic acid and alcohol. The esters of acrylic acid polymerize readily to form exceptionally clear plastics. These are widely used in applications requiring clear durable surfaces, e.g. in the aircraft and automobile industries. In more common use are surface coatings involving acrylics (see articles). The physical properties of the acrylics (such as gloss, hardness, adhesion and flexibility) can be modified by altering the composition of the monomer mixture used in the polymerization process. Acrylics are used in a wide range of industries;

- Adhesives

Both solvent and emulsion acrylic adhesives are extensively used in the industry. A coating must adhere to only one substrate; an adhesive must adhere to one substrate and then to a second substrate. A coating, once applied, is exposed to the elements and must withstand abrasion, marring, solvents, water, and heat. It may require high gloss and other special

properties, as well. An adhesive is protected to a certain degree by being sandwiched between two substrates. It, therefore, does not have to have some of the performance properties that must be built into a coating. It must ideally have a bond strength high enough to fracture or tear at least one of the substrates. In many cases, the bond strength should not be materially affected by heat, solvents, or water. Therefore, an adhesive must not only have good anchorage to both substrates (adhesive strength), it must also have high enough cohesive strength to fracture or tear one of the substrates upon delamination. [34]

Thus, an adhesive must balance adhesive strength with cohesive strength. Another basic difference between emulsions (coatings and adhesives) is in their film formation properties. To have hard, tack-free, and heat-resistant coatings, the glass transition temperature of the polymer is intentionally designed to be higher than room temperature. The coating then requires a coalescing agent to form a clear continuous film. Adhesives form films at room temperature without the need for coalescing aids. A soft flexible polymer film is desired for an adhesive, and this film should be thermoplastic (i.e., able to soften and flow repeatedly upon the application of heat). The film can subsequently be cross-linked through functional groups if heat and solvent resistance are desired. Acrylic-based adhesives are normally employed where improved specific adhesion and/or resistance to yellowing from exposure to ultraviolet rays is required. Acrylics are used in three main areas: heat sealable adhesives, laminating adhesives, and pressure-sensitive adhesives. These are discussed separately. [33]

- The textile industry (e.g. making the sponge fill used in padded jackets)
- Paper coatings
- The paint industry (particularly in paints used for road markings)
- Cement modifiers [34]

2.4.1.2 Versatility of Acrylics

By selecting proper monomers, the glass transition temperature of the polymer and,

therefore, the likely application area, can be varied. The glass transition temperature of a polymer is the simple average value in degrees Celsius representing a range of temperatures through which the polymer changes from a hard and often brittle material into one with soft, rubberlike properties. Although these average Tg values sometimes vary with the test method used, they are reproducible within certain limits and represent specific polymer characteristics. The glass transition temperature is useful as a guideline for softness of hand, low temperature flexibility, and room temperature hardness and softening point.

The glass transition temperature should be used to compare hardness and softness of latex. Table 2.3.1.2a illustrates the wide range in glass temperature (Tg) resulting from different monomer compositions. [35]

Table 2.3 Glass Transition Temperature [35]

Homopolymer	Tg(^{O}c)
Acrylic acid	112
Methyl acrylate	8
Ethyl acrylate	-24
-Butyl acrylate	-56
Acrylic acid	112
Methyl acrylate	8
Ethyl acrylate	-24
-Butyl acrylate	-56
Methyl methacrylate	106

Table 2.4 Glass Transition Temperature versus Application Area [35]

*T*g(°C)	Suggested Application Area
80-100	High heat-resistant coatings
50-65	Floor care coatings
35-50	General industrial coatings
10-40	Decorative paints
25-35	Binders for inks
60-25	Adhesives

2.4.1.3 Emulsion Acrylics

Emulsions have become the dominant technology in acrylic polymers. Table 4.1b correlates T g ranges of emulsion acrylics with specific application areas. There is overlap to be expected among the ranges. There is a clear indication of the versatility of acrylics because of varying monomers. The wide range for adhesives dry to a tack-free state includes pressure-sensitive polymers and heat-activating polymers. [35]

2.4.2 Polyurethane Coating

Polyurethanes are group of plastics that may be either thermosetting or thermoplastic.

Polyurethane can be made into both flexible and rigid foams. The flexible foam is often used in furniture and automobile cushions, in mattresses, and for carpet backings. The rigid foam is used for the thermal insulation of refrigerators, trucks, and buildings. In the furniture industry the rigid foam is molded into mirror frames, chair shells, and other parts that were formerly made from wood. Some polyurethanes are highly elastic materials that are resistant to chemical attack and to abrasion.

They are used in such things as solid rubber tires and shoe heels. Lycra, a fiber used in stretch clothing, is polyurethane. Polyurethanes are also used as decorative and protective coatings, exhibiting high gloss, hardness, and toughness. [31]

Polyurethanes have been developed to be the most extensive and versatile class of polymers since their invention in 1937, most polyurethanes physical properties is based on a segmental primary structure $(A - B)_n$. Segmental polyurethane consists of at least three components which is the soft and the hard segment. They differ in hardness, flexibility, polarity, compatibility and interchange interaction. Thermoplastic polyurethane elastomer in the soft segment is greater than 50% by weight, forming a continuous matrix, in which the hard segment aggregate to micro domains with high cohesion. Micro domains are physical cross-linking sites with reversibly at melting temperature of the hard segments domains are responsible for thermoplastic character. [34]

Most polyurethane has a segmental structure before the transformation into an ionomer. Whether the ionic site is within the soft segment or within the hard segment, there is a substantial difference. Polyurethanes, also known as polycarbamates, belong to a larger class of compounds called polymers. Polymers are macromolecules made up of smaller, repeating units known as monomers. Generally, they consist of a primary long-chain backbone molecule with attached side groups. Polyurethanes are characterized by carbamate groups ($-NHCO_2$) in their molecular backbone. A urethane linkage is produced by reacting an isocyanate group, -N=C=O with a hydroxyl (alcohol) group, -OH. Polyurethanes are produced by the polyaddition reaction of a polyisocyanate with a polyalcohol (polyol) in the presence of a catalyst and other additives. Where a polyisocyanate is a molecule with two or more isocyanate functional groups, R-(N=C=O)n $\geq$ 2 and a polyol is a molecule with two or more hydroxyl functional groups, R'-

(OH)n ≥ 2. The reaction product is a polymer containing the urethane linkage, -RNHCOOR'-. In Europe the meanings for 'A-side' and 'B-side' are reversed. Resin blend additives may include chain extenders, cross linkers, surfactants, flame retardants, blowing agents, pigments, and fillers. [35]

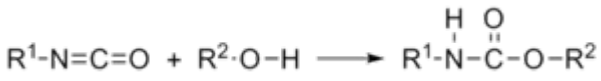


Fig. 2.25 Generalized polyurethane reaction

Polyurethane foams are available in various qualities, such as different densities, FR properties, different porosity and other specialist chemical and physical properties. Polyurethane provides a smooth durable finish that has superior resistance to corrosion, abrasion, and chemical exposure. Polyurethanes are widely used in coatings, flexible and rigid foams, elastomer and composites. Another reason for the use of polyurethane coatings include high performance characteristics such as flexibility, toughness, strength abrasion resistance; chemical resistance such as water, detergent, many industrial chemicals and stain resistance, good light stability when aliphatic isocyanates are used; and low temperature-cure characteristics. [36]

However some uses include leather coatings, fabric coatings and adhesives, industrial maintenance and corrosion resistance finishes, floor vanish, seamless flooring, marine finishes, magnet wire coating and concrete sealing. Urethane coatings are classified as belonging to one of five ASTM classifications, which are chiefly related to the curing mechanism:

A. Oil modified. Isocyanates and alcoholysis products of drying oils. The process is analogous to the one used in alkyd manufacture because they produce an upgraded drying oil. Curing is done by oxidation of the unsaturated oil.
B. Moisture cure. Isocyanates are polymerized with diols and triols such as polyethers and castor oil products. These prepolymers are designed with unreacted terminal isocyanate groups that react with atmosphere moisture to the finished cross-linked polymer film

C. Blocked. Two component systems; the adduct is blocked by reaction with certain polyols, most commonly phenol, making it unreactive at room temperature. It can be packed with polyols, pigments and additives. Heating to temperatures of 121°C or higher unblocks the adduct, producing the ingredients of a two-package system.
D. Prepolymer plus catalyst. They are the same as those mentioned in number B but with separate catalyst to accelerate the cure.
E. Two component. Isocyanates are reacted with relatively low molecular weight polyols such as alcohols to form adducts These adducts then form one part of two-can system; chain extension and curing are obtained from the polyol components. [34]

Polyurethane foam has always been the most extensively used polyurethane; being used in automobile seats, mattresses and insulation for homes, buildings, industrial tanks and piping. Polyurethane elastomers, both thermoset (liquid castable) and thermoplastic, have been used as wheels for forklift trucks, industrial rolls and belting, hose jackets, abrasion resistance gaskets, seals and gears and potting compounds for the telephone and medical industries. [35]

2.4.2.1. Applications and uses of polyurethanes

In modern life polyurethanes are found just about everywhere; the chair used to sit, the bed used to sleep in, the house used to live in, the car used to drive and all these, plus innumerable other items you use every day, probably contain polyurethanes. This section explores some of the more common applications of polyurethanes and provides an insight into their use. It shows how versatile they are and also how they help to protect the environment by conserving energy.

Common polyurethane applications include: Building insulation- Polyutherane is applied as an insulator in buildings. Polyurethanes are regarded as an affordable, durable and safe way of reducing carbon emissions that lead to global warming. They can dramatically reduce heat loss in

homes and offices in cold weather. They play an important role in keeping buildings cool, which means air conditioning can be turned down during the summer. [36]

Refrigerators and freezers - Rigid polyurethane insulating foam makes a major contribution to sustainability and eco-design by reducing the energy required to keep refrigerators and freezers cold. Almost all refrigerators and freezers and most refrigerated trucks produced worldwide are insulated with polyurethane foams.

Furniture and bedding - Flexible polyurethane foams are soft, yet provide good support, durable, and maintain their shape. They are an excellent and safe filling material for seating cushions and mattresses.

Footwear - Light but highly abrasion-resistant polyurethanes are perfect for hardwearing shoe soles, with excellent long-term mechanical properties. Polyurethane soles are practical and keep water out, while in no way limiting design potential.

Coatings and adhesives - The durability, corrosion resistance and weather resistance of polyurethanes make them suitable for coating all kinds of surfaces. Applications range from concrete constructions like bridges and motorway structures, to steel railway carriages and wooden furniture. [35]

Polyurethanes are so versatile that they are also available in the form of glues that can safely bind together quite different materials, such as wood, rubber, cardboard or glass.

Automotive - Polyurethanes are widely used in car manufacture, offering real benefits in terms of comfort, protection and energy conservation. Polyurethane foams can be found in seats, armrests and headrests of most cars, where their cushioning properties help to reduce the fatigue and stress often associated with driving. Their durability and light weight, combined with their strength, make them ideal not only for cushioning purposes, but also within the bodies of cars, where their insulation properties provide protection against the heat and noise of the engine. Because polyurethanes are so strong and light, their use means that the overall weight of cars is

reduced, resulting in greater fuel efficiency and improved environmental performance. Polyurethanes are often applied as lightweight foams which help reduce the weight of cars, thereby lowering fuel consumption and carbon dioxide emissions. [36]

2.4.2.2 Flame Retardant Polyurethane Coatings

For various applications in cars, home appliances, electrical and other industries coating increasingly need to meet flame retardancy requirements. Especially severe restrictions on flammability have been imposed on coating materials for building interior applications. The development of flame retardant polyurethane coatings has received significant attention, because polyurethanes are one of the major polymers used in the coating industry. They are preferred for many applications due to their excellent properties such as good adhesion, abrasion resistance, weather and chemical resistance. They can be formulated to have rapid drying time and long pot life. Bhandari and Chandra proposed flame retardant polyurethane coating by introducing chlorine compounds into a castor oil alkyd. When examining properties of flame retardant, two component polyurethane coatings using various halogens compounds. [37]

One limitation of these halogens containing flame retardant coatings is the problem of toxic gas generation during combustion of halogens compounds. Among non-toxic flame retardant coatings, phosphorus–containing coatings are one possible candidate for the replacement of halogen-containing coatings. It also has been generally accepted that phosphorus containing coating materials is superior to halogen-containing ones in flame retarding performance. The purpose is to synthesize non-toxic reactive polyurethanes and to characterize the physical properties and the flammability of coatings. The increasing public awareness of their potential as fire hazards has stimulated the development of fire retardant polyurethane coating systems. As with other polymeric materials, the flame resistance of polyurethane can be improved by incorporating either reactive or nonreactive fire retardants. [4]

Flame retardants can be classified as organic materials. Even though inorganic flame retardants offer low cost and a lack of toxic by-products, their effectiveness is relatively poor. High level of additives is required to reach the desired flame retardant levels. Organic

phosphorus-containing compounds are believed to be very effective nontoxic flame retardant additives in lowering the pyrolytic decomposition and temperature of the polymer while raising the yield of incombustible char. This is based on formation of phosphorus acid, which promotes the formation of char to protect the polymer from the flame and heat. [37]

The conventional solution-based coatings are of two types; one component systems and two-component systems.

2.4.2.3 One component system

One-component systems have two types: reactive and completely reacted systems.

- Reactive one-component systems: these systems are low molecular weight pre-polymers with terminal isocyanate groups. They are dissolved in solvents of low polarity. After coating, they are moisture cured. The water acts as a chain extender and cross-linking agent with the formation of urea and biuret linkages.
- The generation of carbon dioxide is sufficiently slow, so that slow diffusion of the gas from the film occurs without bubble formation. The rate of cure is dependent on the temperature of the cure and the humidity of the ambient. Use of blocked isocyanate pre-polymers allows formulations of one component systems that are stable at room temperature.
- Completely reacted one-component system: this consists of totally reacted high molecular weight thermoplastic polyurethane elastomers. The PU is dissolved in a highly polar solvent like dimethyl formamide. These coatings dry physically. [36]

2.4.2.4 Two component system

Isocyanate-terminated pre-polymers or polyfunctional isocyanates are reacted with polyhydroxy compounds that may already be urethane modified. The polyisocyanate component, usually in the form of a solution, is mixed with the polyhydroxy component prior to coating. Curing of these coatings occurs due to the formation of urethane linkages.

In addition, reaction with moisture also takes place. The properties of the resulting coatings depend on various factors

(A) The polyol type and molecular weight

(B) The temperature of the reaction

(C) The concentration of polar groups, i.e., urethane and urea

(D) The cross-linking density [36]

2.4.2.5 Additives for urethane coatings

The additives used for urethane coatings are generally silica fillers to reduce gloss, UV absorbers, antioxidants, and flow improvers. The solvents used for coating should be free of moisture and reactive hydrogen to prevent reaction with free isocyanate in two-component systems. They are generally polar in nature, and care should be taken for their selection to ensure storage stability and blister-free film. Similarly, the pigment used should also be moisture free. [37]

2.5 Testing methods for flammability

Differing performance requirements and government regulations have led to the development of numerous test methods for evaluating the flame retardancy of textiles. There are numerous test methods with vertical, horizontal or diagonal arrangement of the samples, methods with and without air ventilation, and many special tests. [3]

Table 2. 5 Common flammability test

Test method	Sponsoring organization	Comments
16 CFR 1610	Consumer Product Safety Commission (CPSC) 1 s	Fabric at 45º angle to flame for 1s. For general apparel.
16 CFR 1615/1616	CPSC	Fabric held vertical to flame for 3 s. For children's sleepwear.

NFPA 1971	National Firefighters Protection Association (NFPA)	Fabric held vertical to flame for 12s. . For protective clothing.
NFPA 701	NFPA	Fabric held vertical to flame for 45 s to 2 min. For drapery.
ASTM D-2863 Limiting oxygen index (LOI)	ASTM	Fabric is held vertical in atmosphere of different oxygen/ nitrogen ratios and ignited from top. Determines minimum oxygen level to support combustion.
BS 5852 Part 1 and 2, ignition sources 'cigarette' and 'match' equivalent also EN 1021 and EN 597	British Standards Institution	Burning behavior of for upholstered furniture fabrics (also for private use) against smoker-materials like cigarettes and matches. Finished fabric must be soaking resistant at 40 °C according to BS 5651, then horizontally and vertically fixed on a mini chair on a support of foamed PU, by seven ignition methods.
ISO 6940/6941	International Standards Organization	Vertically held specimens, determination of the ease of ignition/the flame spread properties.
DIN 54333 T1	Deutsches Institut für Normung	Horizontally held specimens, because of the heat distribution less severe than vertical tests.

Table 2.6 Durably-finished and inherently flame-retardant fibres in common use

Fibre	Flame-retardant structural components	Mode of introduction
Natural:		

Cotton	Organophosphorus and nitrogen-containing monomeric or reactive species – e.g. Proban CC (Rhodia, formerly Albright and Wilson), Pyrovatex CP (Ciba), Aflammit P and KWB (Thor), Flacavon WP (Schill and Seilacher)	F
	Antimony–organohalogen systems – e.g. Flacavon F12/97(Schill and Seilacher), Myflam (Noveon, formerly Mydrin)	F
Wool	Zirconium hexafluoride complexes – e.g. Zirpro (IWS) Pyrovatex CP (Ciba), Aflammit ZR (Thor)	F
Regenerated:		
Viscose	Organophosphorus and nitrogen/sulphur-containing species – e.g. Sandoflam 5060 (Clariant, formerly Sandoz) in FR Viscose (Lenzing) Polysilicic acid and complexes – e.g. Visil AP (Sateri)	A
Synthetic		
Polyester	Organophosphorus species: phosphinic acidic comonomer – e.g. Trevira CS (Trevira GmbH, formerly Hoechst), phosphorus-containing additive, Fidion FR (Montefibre	C/A

Continue **Table 2.6**

Durably-finished and inherently flame-retardant fibres in common use

Fibre	Flame-retardant structural components	Mode of introduction
Polypropylene	Halo-organic compounds usually	A

	as brominated derivatives – e.g. Sandoflam 5072 (Clariant, formerly Sandoz)	
Inherent :		
Polyhaloalkenes	Polyvinyl chloride – e.g. Clevyl (Rhodia, formerly Rhone-Poulenc) Polyvinylidene chloride – e.g. Saran (Saran Corp.)	H
Polyaramids	Poly (*m*-phenylene isophthalamide) – e.g. Nomex (Du Pont), Conex (Teijin)	Ar
	Poly (*p*-phenylene terephthalamide) – e.g. Ar Kevlar (Du Pont), Twaron (Enka)	Ar
Poly (Aramid–Arimid)	For example: Kermel (Rhodia)	Ar
Polybenzimidazole	For example: PBI (Hoechst-Celanese)	

Key:

F: chemical finish

A: additive introduced during fibre production

C: copolymeric modifications

H: homopolymer

Ar: aromatic homo- or copolymer

2.5.1 Limiting Oxygen Index

The evaluation of fire retardancy is carried out by a variety of techniques, most of which do not correlate well with other test protocols. The three most common methods that are used are the oxygen index, the UL-94 test, and cone calorimetry. Oxygen index is an evaluation

of the ease of extinction of a fire, how rapidly does the flame chemistry lead to extinction. The measurement consists of determining the minimum concentration of oxygen in a nitrogen–oxygen mixture that will sustain combustion. A measure that enables an obvious assessment of flame protection properties is the limiting oxygen index (LOI) (fig 2.26) , determined according to ASTM D-2863. The LOI is defined as the content of oxygen in an oxygen/nitrogen mixture that keeps the sample at the limit of burning: LOI = 100 × O2 : (O2 + N2).

As the oxygen content of air is 20 % corresponding to LOI = 20 all textiles with lower LOI values will burn quite easily in air and those with LOI values much higher than 20 will not burn. For all methods, strict adherence to the testing protocol is crucial to obtaining reliable and repeatable results. However, it is important to recognise that if a fabric passes a particular test, it just means that the fabric passed this particular test. There are no other performance guarantees. More detailed information on actual flame retardancy test methods and an outlook on their development have been published. Oxygen index is an evaluation of the ease of extinction of a fire, how rapidly does the flame chemistry lead to extinction. The measurement consists of determining the minimum concentration of oxygen in a nitrogen–oxygen mixture that will sustain combustion. [38]

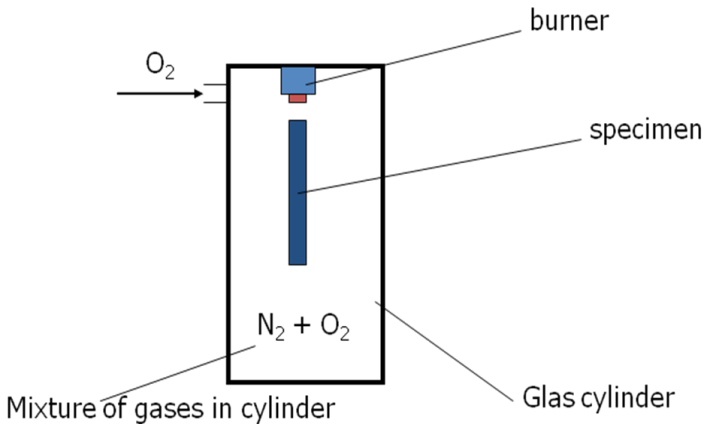


Fig. 2.26 Limiting Oxygen Index

Limiting oxygen index (LOI) is a measure that enables an obvious assessment of flame,

protection properties are the determined according to ASTM D-2863. The LOI is defined as the content of oxygen in an oxygen/nitrogen mixture that keeps the sample at the limit of burning: LOI = 100 × O2: (O2 + N2). As the oxygen content of air is 20 % corresponding to LOI = 20 all textiles with lower LOI values will burn quite easily in air and those with LOI values much higher than 20 will not burn. [11]

The burning behaviour of above fibres is influenced by a number of thermal transition temperatures and thermodynamic parameters. Listed in Table 2.6 are fibres with their physical glass (Tg) and melting (Tm) transition temperatures. The lower the respective *T*c (and usually *T*p) temperature and the hotter the flame, the more flammable is the fibre. Limiting Oxygen Index (LOI) values are listed, which are measures of the inherent burning character of a material.

Under real fire conditions, it is the rate of heat release that determines burning hazard and heats of combustion indicate that little difference exists between all fibres. Cotton has low heat combustion and textiles used in building materials and aircraft and transport seating require having minimal levels of rate of heat release and is measured in instrument called cone calorimeter. [38]

3 Experimental part

3.1 Aim of the experiment

The aim of the experiment was to compare impregnation and coating methods of standard ecological flame retardant finish on polyester and polyester-cotton blends

To study the flammability of coated and impregnated textiles by different tests, simulate different orientation of textile at burning.

To study thermal capacity of textile with no coating or impregnation and also with coating or impregnation, by the use of differential scanning calorimetry, laser treatment and other supporting tests.

3.2 Description of materials used

Material used in the experiment 100% polyester twill weave and the square weight 441g/m^2 provided by Inotex spol. s r.o., Dvů Králové n/L. Blended 65% polyester/35% cotton twill weave with the square weight of 458g/m^2 also provided by Inotex spol. s r.o., Dvů Králové n/L.

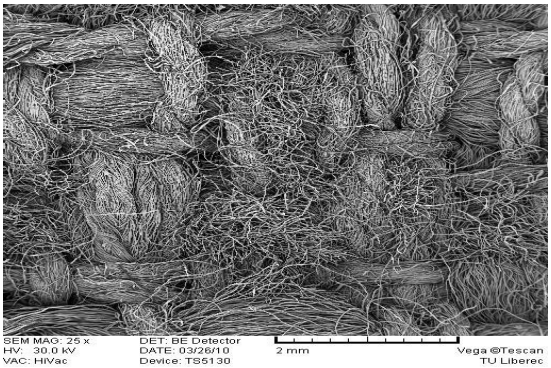


Fig. 1 Structure of 100 % polyester twill twill weave (face)

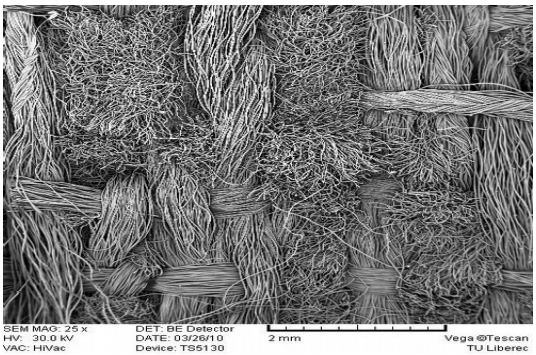


Fig. 2 Structure of 100 % polyester twill weave (back)

100% Polyester twill weave yarn count:

Warp: 35.8 tex

: Sett 35.9 yarn/cm

Weft: strong thread lighter is 466 tex,

: Strong thread darker is 461 tex,

: Thin thread is 36.9 tex.

: Sett 11.9 yarn/cm

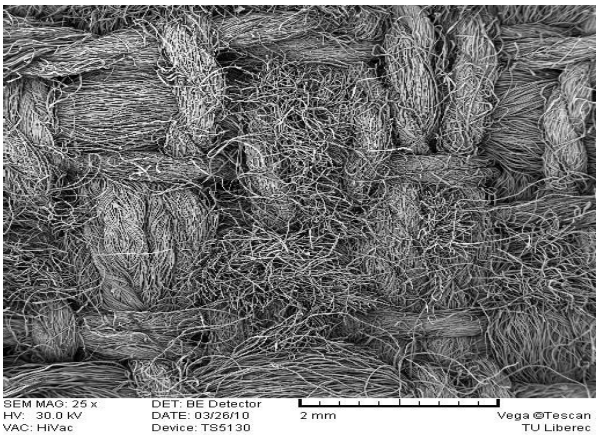


Fig. 3 Structure of 65% PE / 35% CO twill weave (face)

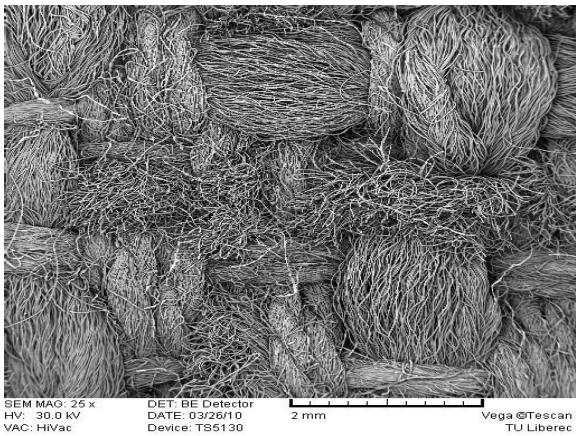


Fig. 4 Structure of 65% PE / 35% CO twill weave (back)

Blended 65% polyester /35% cotton twill weave yarn count:

Warp : strong thread lighter: 213tex,

: Strong thread darker: 461 tex,

: Thin thread: 54.1 tex,

: Sett 27.8yarn/cm

Weft: strong thread lighter is 227 tex,

: Strong thread darker is 349 tex,

: Thin thread is 50.3 tex,

: Sett 13.4 yarn/cm

3.3 Used Fire Retardant Products

In this thesis, the following products used were produced by Inotex spol. s r.o., Dvůr Králové n/L, which is characterized by:

Texaflam PE Concentrated: Wash permanent fire retardant applied by impregnation. It is the mixture of phosphonate complexes, viscous liquid and non-ionic. Texaflam PE concentrated is also an agent for permanent flameproof finish on 100 % polyester applied by Thermosol process. It is also suitable for treatment clothing and technical fabrics, decorative & curtain fabrics. FR effect is resistant to water, shampooing, dry-cleaning, and washing up to 25 x 40°C.

Texaflam BS: Agent for semi permanent flameproof finishing of cellulosic and synthetic materials (PET, PAD, PAN) and blends. It is suitable especially for furniture and upholstery fabrics. Semi permanent FR-effect is ensured by polymeric film-forming agent resulting in adhesivity of FR component on the fibre. FR effect is resistant to water, shampooing, and dry-cleaning.

Texaflam XPE: Coating agent for semi-permanent flameproof finish of cellulosic (cotton, viscose) and synthetic materials (PET, PA, PAN) and blends. Especially for furniture and upholstery fabrics: woven textiles and knits. Synthetic polymers emulsion containing Sb_2O_3/Br. Application by back coating (foam or paste).

Texaflam XPE ECO: Ecofriendly phosphate-based halogen free, antimony free and and formaldehyde free FR back-coating system determined mainly for fabrics from natural fibers and their blends with synthetics. It is suitable mainly for upholstery fabric for domestic and automotive applications. It is application by a paste technique - back coating.

3.4 Procedure for the experiment

A recipe for Texaflam PE Concentration

200g/l Texaflam PE Concentrated,

1 gl^{-1} soda (for pH = 6.3),
3.0 gl^{-1} TEXAPAL AA

100 % polyester and blended 65% polyester /35% cotton twill weave

In the experimental part the methodology knife over roll on laboratory machine Werner Mathis CH – 8155 from Inotex Company was used for the coating application and impregnation. Two strips of 100% polyester twill weave fabric twill that were cut were impregnated in a bath containing 200g/l Texaflam PE Concentrated, 1g/l soda (for pH = 6.3), 3.0 g/l Texapal AA (wetting agent for lower absorptive materials and better uniformity). The wet pick-up minimum was 40%. The speed of the fabric was 0.77m s^{-1} and dried for 2minutes at 110-135°C, in a Werner Mathis CH – 8155. Curing was done at 180°C for 1 minute and for knits is 30 seconds at 190°C.

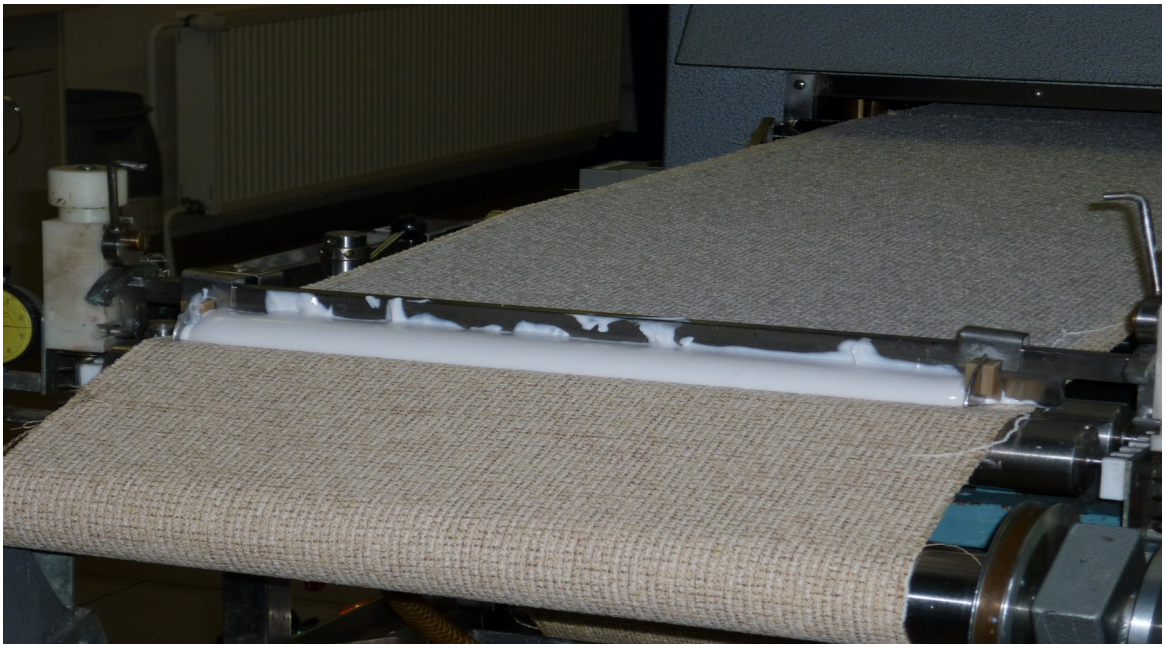


Fig 3.1 Back-coating of Polyester twill on a Werner Mathis CH – 8155 from Inotex spol. s r.o.,

On two strips of 65% PES/ 35% cotton twill weave were impregnated with the same bath as 100% polyester twill weave which means a bath containing 200g/l Texaflam PE Concentration, 1g/l soda (for pH = 6.3), 3.0 g/l Texapal AA (wetting agent for lower absorptive materials and better uniformity). The wet pick-up minimum was 47%. The speed of the fabric was 0.77m s^{-1} and dried

for 2minutes at 110-135°C, in a Werner Mathis CH – 8155. Curing was done at 180°C for 1 minute. Texaflam PE can be stored for minimum of 6 months at temperatures up to 30°C in closed containers. It is a clear highly viscous liquid. The product specific weight is 1.27 $g.ml^{-1}$(25°C) and pH ranges from 1.8-2.6.

It is also nonionic soluble in cold and warm water in any ratio. Compatible with most textile auxiliaries and chemicals nevertheless should be verified before use. Handle of treated goods is generally not influenced. Color shade is not influenced at mass dyed goods. In other cases shade should be changed in dependence on the dyes used. This is recommended to be tested in advance and modify the dyeing bath formulation if necessary. Compatible with Optical Brightening Agents (one-bath application). Hydrophobic or oleophobic finishes can be applied in one bath or in two-step processing in dependence on substrate quality.

A recipe for Texaflam BS Concentration

500 gl^{-1} Texaflam BS,
1 gl^{-1} TEXAPAL AA,

100 % polyester and blended 65% polyester /35% cotton twill weave

In this experiment two 100% polyester twill weave strips were impregnated in a bath containing 500 g/l Texaflam BS, 1g/l Texapal AA (wetting agent for materials with lower absorptivity) and wet pick-up was a minimum of 53%. The speed of the fabric was 0.38m s^{-1} and dried for 2minutes at 100°C, in a Werner Mathis CH – 8155. Curing was done at 150°C for 3 minute and can also be cured at 170°C for 1 minute.

Fig 3.2 Fabric in a Werner Mathis CH – 8155 from Inotex spol. s r.o.,

Also 65% PES/ 35% cotton twill weave were impregnated with bath containing 500 g/l Texaflam BS, 1g/l Texapal AA and wet pick-up was a minimum of 60%. The speed of the fabric was 0.38m s^{-1} and dried for 2 minutes at 100°C, in a Werner Mathis CH – 8155. Curing was done at 150°C for 3 minute. Texapal AA can be stored for minimum of 6 months at temperatures up to 25°C. Frozen product can be used in a conventional way after thawing (at cca 20°C) and stirring. Texaflam BS is slightly yellowish clear viscose liquid and easily soluble in water in any proportions.

Density cca 1.2 $g.cm^{-3}$ and slightly anionic. It is formaldehyde-free, heavy metal-free, halogen-free, Sb_2O_3-free and also inherent antistatic properties.

A recipe for Texaflam XPE ECO(SB/Br – free) paste

100 % polyester and blended 65% polyester /35% cotton twill weave

The coating paste was gauged on the head coating area to the knife and as the fabric was moved from the head area, Texaflam XPE ECO (SB/Br – free) coating paste was coated on the fabric surface .Texaflam XPE ECO (SB/Br – free) coating was applied on two strips of 100%

polyester twill weave by back coating see fig 3.1 and dry wet pick-up 23%.

Also 2 strips of 65% PES/ 35% cotton twill weave were coated by Texaflam XPE ECO (SB/Br – free) and dry wet pick-up was 30%. After coating application, four substrate with paste on the surface were drawn in the heating part and finally wound on the winding roller. The speed of the fabric in the machine was 0.23m s^{-1} while drying at 130°C for 5 minutes and the pressure was 20kPa.

From the four strips coated with Texaflam XPE ECO (SB/Br – free), two strips were given 2nd coat of the same product (100% polyester and 65% PES/ 35% cotton twill weave) on floating knife. The fabrics were dried at 130 °C for 5 minutes and the speed of the fabric in the machine was 0.23m s^{-1}. Fixation at 150°C for 3 minutes and the speed for fixation of the fabric in the machine was 0.38m s^{-1}.

Standards: BS 5852, EN 1021-1, EN 1021-2 (soaking tests, match and cigarette equivalent ignition source). Texaflam XPE ECO (SB/Br – free) is white paste, transparent film after application and aqueous synthetic polymer emulsion. It is miscible in water in any proportions, slight ammonia odour. The density cca 1.15g.cm^{-3} and pH is approximately 6.5. Viscosity (Brookfield RVT Spindle 7, 100rpm): 90 poise antimony and halogen-free also formaldehyde-free. Film properties it gives a soft hand and about 55% dry content. The product can be stored for minimum of 6 months at temperature above 0°C.

A recipe for Texaflam XPE (SB/Br – free) foam

250 gl^{-1} Texaflam XPE (SB/Br – free) foam,
500 gl^{-1} Texaflam XPE (SB/Br – free) foam

100 % polyester and blended 65% polyester /35% cotton twill weave

A 100% polyester and 65% PES/ 35% cotton were coated by 250g/l foam coating contained Texaflam XPE (SB/Br – free) and dried at 130°C for 3 minutes and the speed was

0. 38m s^{-1}. Fixation was done at 150°C for 1 minute with the speed of 1.15m s^{-1}.

Lastly, 100% polyester and 65% PES/ 35% cotton were coated by 500g/l foam Texaflam XPE (SB/Br – free) coating and dried at 130°C for 3 minutes and the speed was 0. 38m s^{-1}. Fixation was done at 150°C for 1 minute with the speed of 1.15m s^{-1} with the pressure of 20kPa.

4. Results

Fire-retardant properties of original and finished fabrics were estimated by using LOI-value and flammability tests determinations. The mechanism of flame retardancy was also studied with DSC technique and burning with laser. The test results of 100% polyester twill weave studies are compared with the results of 65%polyester/35cotton twill weave studies.

Also air permeability, breathability, color difference and tensile strength were tested as supporting tests.

4.1 Flammability testing of textiles and its pretreatment

Assessment of the irritability of upholstered furniture – Part 1: Ignition source shouldering cigarette EN 1021-1(2/2006)

TABLE 4. 1 Description of 100% polyester and it's pretreatment

The dimensions of test specimen :	650 x 800 mm
Test rig assembly :	foam with reduced flammability, 75 mm, Duren KF 2530 + test sample
The pretreatment of the specimen :	water soak before conditional
Test conditions: - Conditioning of test specimens and ignition source :	t = 23 ± 2°C 50 ± 5% r.h.

- Test temperature and rel. humidity :	t = 23 ± 2°C 65±4 % r.h.

Texaflam BS: Standards: BS 5852, EN ISO 1021-1, EN ISO 1021-2 (soaking tests, match and cigarette equivalent ignition source).

Assessment of the irritability of upholstered furniture – Part 2: Ignition source match flame equivalent EN 1021-2(2/2006)

TABLE 4.2 Description of 65% polyester/ 35% cotton sample and it's pretreatment

The dimensions of test specimen :	650 x 800 mm
Test rig assembly :	foam with reduced flammability, 75 mm, Duren KF 2530 + test sample
The pretreatment of the specimen :	water soak before conditional
Ignition :	15 s
Test conditions:	
- Conditioning of test specimens and ignition source :	t = 23 ± 2°C 50 ± 5% r.h.
- Test temperature and rel. humidity :	t = 23 ± 2°C 65±4 % r.h.

TABLE 4.3 Experimental results of flammability of 100% polyester twill weave

Sample	LOI /%/ warp / weft	Square Weight (g/m^2)	Air Permeability (mm/s)	Breathability ($g/m^2.Pa.h$)
Original	21.9 / 19.8	441	343.2	0.18

Sample	1021-1	1021-2	ISO 3795*	LOI /%/ warp / weft	Square Weight (g/m^2) (dry pick-up %)	Air Permeability (mm/s)	Breathability ($g/m^2.Pa.h$)
PE Conc.	PASS	PASS	Length of burning: 0mm Time of burning: 0s Speed of burning: 0mm/min in longitudinal	34.0 / 30.8	593 (+34.47%)	266.4	0.23
BS	PASS	PASS		27.0 / 27.1	555 (+25.85%)	275.6	0.28
XPE 250 g/l	PASS	PASS		28.7 / 28.7	522 (+18.37%)	306.4	0.22
XPE 500 g/l	PASS	PASS		28.1 / 28.4	539 (+22.22%)	268.9	0.22

XPE ECO 1x	PASS	PASS	and transversal directi	24.6 / 24.7	576 (+30.61%)	151.1	0.15
XPE ECO 2x	PASS	PASS		26.5 / 25.8	648 (+46.94%)	72.7	0.10

*time of ignition: 15s; sample size 356x100cm

** results are all the same for flammability test using the BS EN 1021–1/1021-2

Conditions of testing: RH: 65±4%; temperature: 20±2°C

Air permeability EN ISO 9237: pressure drop: 200Pa, sample size: 20cm^2

Breathability EN ISO 15496: TA=TB=23°C, delta p= 2168Pa

TABLE 4.4 Experimental results of flammability of 65% polyester / 35% cotton twill weave

Sample	LOI /%/ warp / weft	Square Weight (g/m^2)	Air Permeability (mm/s)	Breathability (g/m^2.Pa.h)
Original	18.8 / 18.9	458	273.9	0.23

Sample	1021-1	1021-2	ISO 3795*	LOI /%/ warp / weft	Square Weight (g/m^2) (dry pick-up %)	Air Permeability (mm/s)	Breathability (g/m^2.Pa.h)
PE Conc.	PASS	PASS	Length of burning: 0mm Time of burning: 0s Speed of burning: 0mm/min in longitudinal and	29.2 / 27.3	548 (+19.654%)	152.8	0.23
BS	PASS	PASS		27.0 / 28.7	585 (+27.23%)	192.1	0.21
XPE 250 g/l	PASS	PASS		26.9 / 26.7	569 (+24.24%)	197.1	0.22
XPE 500 g/l	PASS	PASS		25.1 / 25.2	620 (+35.37%)	58.5	0.14
XPE ECO 1x	PASS	PASS		26.5 / 26.8	679 (+48.25%)	22.5	0.09

XPE ECO 2x	PASS	PASS	transversal direction	26.9 / 27.1	590 (+28.82%)	118.6	0.21

*time of ignition: 15s; sample size 356x100cm

** results are all the same for flammability test using the BS EN 1021–1/1021-2

Conditions of testing: RH: 65±4%; temperature: 20±2°C

Air permeability EN ISO 9237: pressure drop: 200Pa, sample size: 20cm^2

Breathability EN ISO 15496: TA=TB=23°C, delta p= 2168Pa. Samples XPE ECO 1x and XPE ECO 2x interchanged according to the dry pick-up and breathability and air permeability.

Finished fabrics properties coated by Texaflam BS have minimum tensile strength loss and slightly increased stiffness mainly at cotton. There is also a risk of yellowing at white materials and also enhancing electro conductivity.

TABLE 4.5 Test results relate only to the ignitability of the combination of 100% polyester under the particular conditions of test

	Cigarette		**Comments**
	1*	2*	
Smoldering criteria			
Unsafe escalating combustion	no	no	The cigarette burnt out, there was no flaming or progressive smouldering.
Test assembly consumed	no	no	The cigarette burnt out, there was no flaming or progressive smouldering.
Smoulders to extremities	no	no	The cigarette burnt out, there was no flaming or progressive smouldering.
Smoulders through thickness	no	no	The cigarette burnt out, there was no flaming or progressive smouldering.
Smoulders more than 1 h	no	no	The cigarette burnt out, there was no flaming or

			progressive smouldering.
In final examination, presence of active smoldering	no	no	The cigarette burnt out, there was no flaming or progressive smouldering.
Flaming criteria			
Occurrence of flames	no	no	The cigarette burnt out, there was no progressive smouldering.
*Enter YES if criteria exceeded or NO if criteria not exceeded.			

The above results are not intended as a means of assessing the full potential fire hazard of the materials in use.

TABLE 4.6 Test results relate only to the ignitability of the combination of 65% polyester / 35% cotton under the particular conditions of test.

	Match flame equivalent			**Comments**
	1*	2*	3*	
Smouldering criteria				
Unsafe escalating combustion	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Test assembly consumed	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Smolders to extremities	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Smolders through thickness	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Smolders more than 1 h	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
In final examination, presence of active smoldering	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Flaming criteria				
Unsafe escalating combustion	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Test assembly consumed	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
Flames to extremities	no	no	no	Flaming ceased after removal of the

				burner, there was no progressive smouldering.
Flames through thickness	no	no	no	Flaming ceased 1 second after removal of the burner, there was no progressive smouldering.
Flames Langer than 120 s	no	no	no	Flaming ceased after removal of the burner, there was no progressive smouldering.
*Enter YES if criteria exceeded or NO if criteria not exceeded.				

Notice: The above results are not intended as a means of assessing the full potential fire hazard of the materials in use

****The supplement:**

BS 5852:2006, section 11 – while testing with smoldering cigarette and match flame equivalent as ignition sources for ignitability of upholstered furniture assessment references to EN 1021-1 and 1021-2 are to be respected

4.2 Discussion of flammability tests

4.2.1 Flammability test of polyester twill weave

Polyesters are subject to serious degradation in the presence of a number of conventional used flame retardants with a loss in physical properties. FR polyester is perceived to be a more suitable material for outdoor work tasks. Polyester without application of fireproof totally melts in high temperature above 265^{o}C. Using BS EN 1021-1/1021-2, the experimental samples pass the flammability test as they did not burn when subjected to flame as shown in Table 4.3. The time of ignition was 15 seconds and the sample size 356x100cm, in conditions of testing RH (relative humidity) was 65+4% at temperature: 20+2°C. Specimens were tested warp and weft using limiting oxygen index and different values were obtained. The use 200gl^{-1} Texaflam PE concentrated gave value of 34.0 warps in which its threads run lengthways of the material and 30.8 weft which runs across from side to side, when tested using limiting oxygen index. Also those were the highest values obtained. 200gl^{-1} Texaflam PE concentrated is permanent fire

retardant that is resistant to water; it forms a protective liquid or char barrier also suitable for polyester. This limits the amount of polymer available to the flame front and acts as an insulating layer to reduce the heat transfer from the flame to the polymer. Foam coating using Texaflam XPE with different concentrations gave almost the same value limiting oxygen index for warp and weft. Antimony trichloride evolves heavy vapours which formed a layer over the condensed phase, stop oxygen attack and thus choke the flame. Also the liquid and solid antimony trichloride particles contained in the gas phase reduce the energy content of the flames.

Limiting oxygen index of 500gl^{-1}Texaflam BS was 27 for warp and 27.1 for weft.

When Texaflam XPE ECO coating was applied twice, limiting oxygen index was higher than when applied once in both warp and weft. Fabric add-on determines the overall performance of fire resistant in coating.

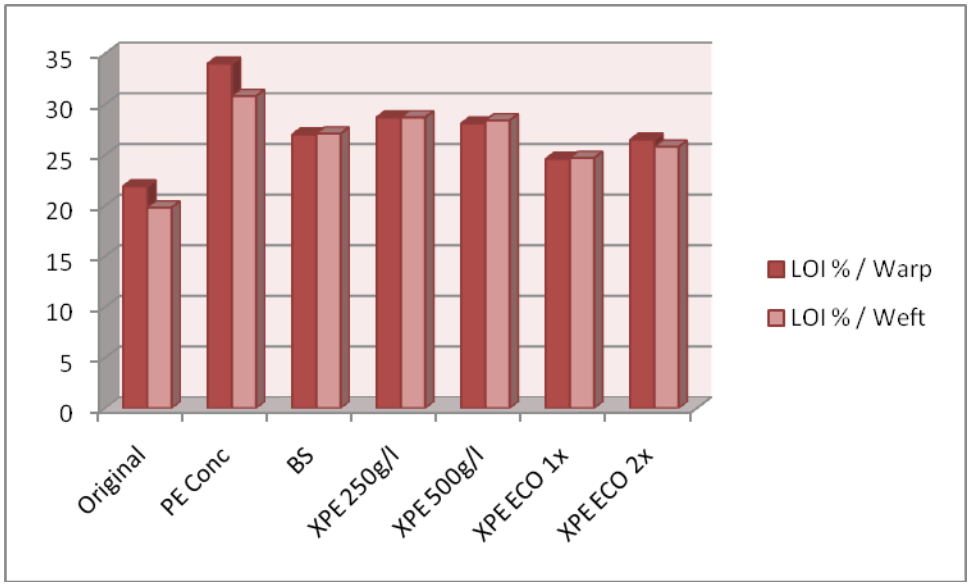


Fig. 4.1 Limited Oxygen Index values of polyester twill weave warp and weft

Thickness of coating layer plays a very important role in permeability and water repellency.

4.2.2 Flammability test of polyester cotton twill weave

Pure cotton is combustible at high temperatures while polyesters tend to shrink away from flames and self-extinguish upon ignition. Pure cotton burns when put on flame and has an afterglow, the smell is like a burning paper and leaves a black and powdered ash. Using BS EN 1021-1/1021-2, the experimental samples pass the test as they did not burn when subjected to flame as shown in Table 4.4.The time of ignition was 15 seconds and the sample size 356x100cm, in conditions of testing RH (relative humidity) was 65+4% at temperature: 20+2°C.

The use $200gl^{-1}$ Texaflam PE concentrated gave value of 29.2% warp and 27.3% weft when tested using limiting oxygen index. Also those were the highest values obtained. $250gl^{-1}$ Texaflam XPE coating gave value of approximately 26% and was better limiting oxygen index than $500gl^{-1}$ Texaflam XPE foam coating with approximately 25% limiting oxygen index. The agent is very good on the blends. Texaflam XPE ECO when applied once and twice gave approximately 26% value of limiting oxygen index. The flame retarding effect is due, in all probability, to the oxidation of elemental phosphorus during the combustion process to phosphoric acid. A carbonaceous layer is formed in the condensed phase. The formation of fragment acts by interrupting the radical chain mechanism.

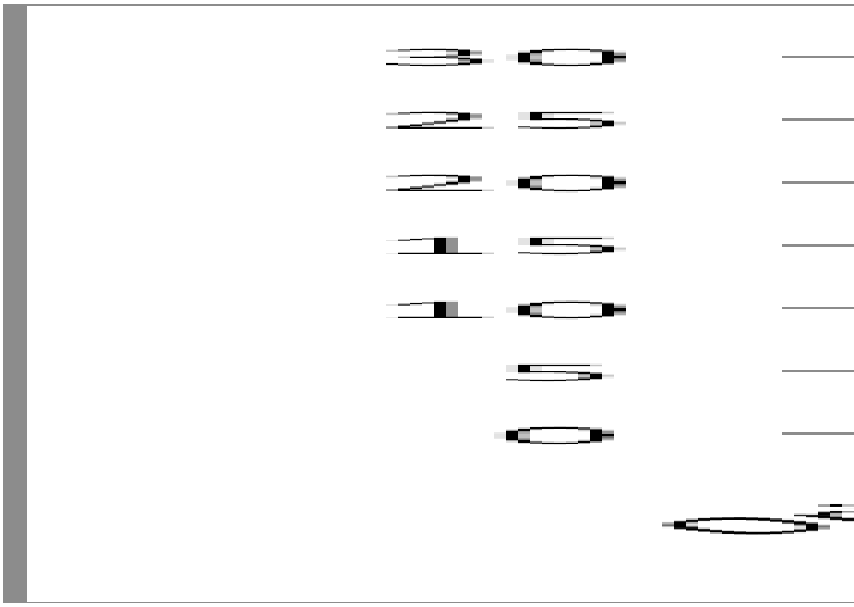


Fig. 4.2 Limited Oxygen Index values of polyester/cotton twill weave warp and weft

4.3 Air-permeability testing of textiles and its machinery

Air permeability is the rate of air flow passing perpendicularly through a known area under a prescribed air pressure differential between the two surfaces of a material of textile fabrics and is applicable to most fabrics including woven fabrics, air bag fabrics, blankets, napped fabrics, knitted fabrics, layered fabrics, and pile fabrics. The fabrics may be treated, untreated, heavily sized, impregnated, coated, and resin-treated. It is generally expressed in SI units as $cm^3/s/cm^2$ but in this diploma work is expressed in mm/s.

Air permeability is an important factor in the performance of such textile materials as gas filter, fabrics for air bags, clothing, mosquito netting, parachutes, sails, and vaccum cleaners. It provides an indication of the breathability of weather-resistant and rainproof fabrics. Different surfaces of a fabric, its texture can have different air permeability. But that depends upon the direction of air flow. Yarn twist is very important for woven fabrics. The yarn diameter and the cover factor are decreased, as twist increases. This increases air permeability. Increasing yarn twist may also allow the more circular, high-density yarns to be packed closely together in a tightly woven structure with reduced air permeability.

4.3.1 Measuring air permeability and discussion of results

The standard test method used was according to EN ISO 9237. The clamping system was loosened by twisting the top ring counter-clockwise. The fabric sample was positioned between the top and bottom of the column, ensuring that fabric covers the entire opening. The clamping system was tightened completely. A circle of fabric was clamped into the tester and through the use of a vaccum. Coated test specimens were placed with the coated side down to minimize air edge leakage. Airflow occurs from the side with higher air pressure, through the fabric, to the side with the lower air pressure. From that rate of air flow, the air permeability of the fabric was determined. For scientific testing, 10 samples were used. The specimens were tested

individually; values used were read directly from the test instrument in SI units. Average air permeability was calculated the average air permeability for the entire sampling unit.

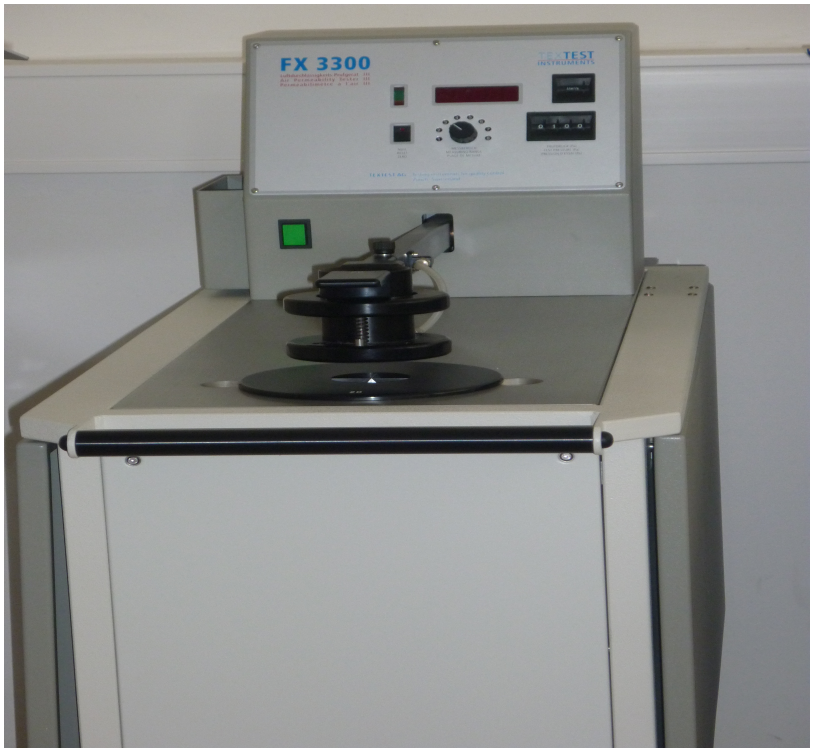


Fig. 4.3 Air permeability testing machine FX 3300

The results of this experiment for 100% polyester twill weave are listed in table 4.3 and for 65% polyester / 35% cotton twill weave in table 4.4. The purpose of the study is to compare air-permeability original, impregnated and coated materials. In fig. 4.4 the air permeability values are shown where 100% polyester twill weave with low air permeability which was coated twice with Texaflam XPE ECO (SB/Br-free). Fabric with low air permeability offers high protection. Foam coating with $250gl^{-1}$ Texaflam XPE (SB/Br-free) offered high air permeability compared to other impregnated and coated samples in fig. 4.4 except original.

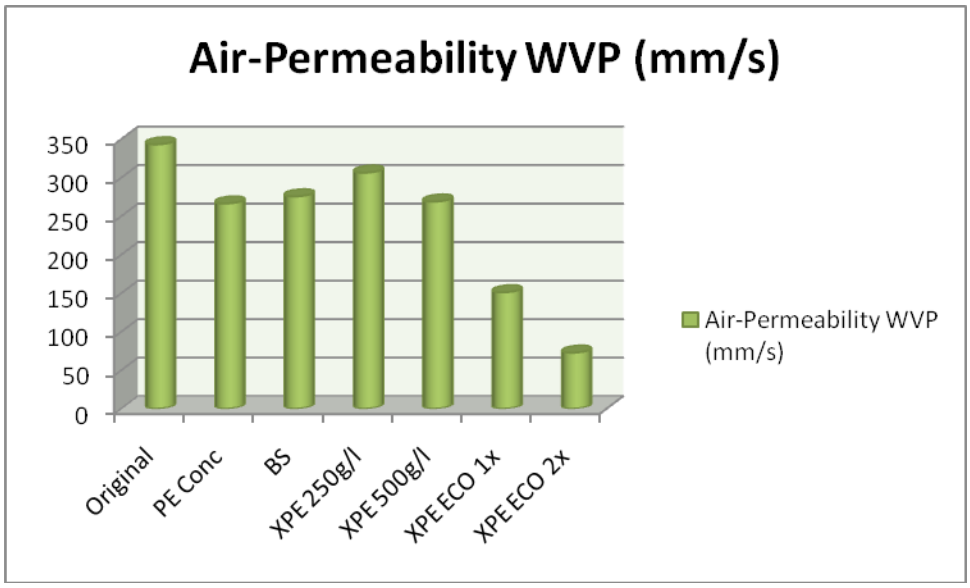


Fig. 4.4 Air- Permeability values of 100% polyester twill weave

In fig. 4.5 where 65% polyester / 35% cotton twill weave coated with Texaflam XPE ECO (SB/Br-free) offered low air permeability, followed by $500gl^{-1}$ Texaflam XPE (SB/Br-free) foam coating. $250gl^{-1}$ Texaflam XPE (SB/Br-free) foam coating offered high air permeability again. High air permeability offers a low protection. This is because an increase of the air permeability results in an increase of the air velocity through the fabric. The air-permeability of material decrease after impregnation and coating.

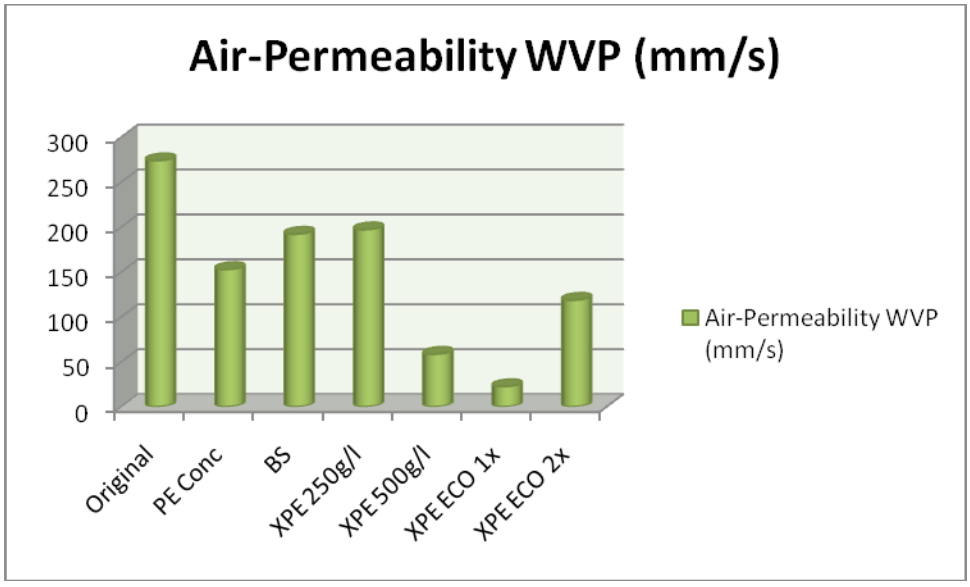


Fig. 4.5 Air- Permeability values of 65%polyester/35% cotton twill weave

4.4 Breathability of the textile and its machine

This is the measuring of water vapor concentration calculating how much water vapor crosses the sample. Results may be shown in terms of water vapor flux (grams/square meter/day) or resistance to the diffusion of water vapor (units of s/m). The lower the diffusion resistance, the more water vapor gets through the material. The reason for doing the testing this way is that some materials like have much better water vapor transports properties when they are in a humid environment than when they are in a dry environment. Other materials, such as most textiles or micro porous membranes, have a nearly constant water vapor diffusion resistance regardless of the environmental conditions

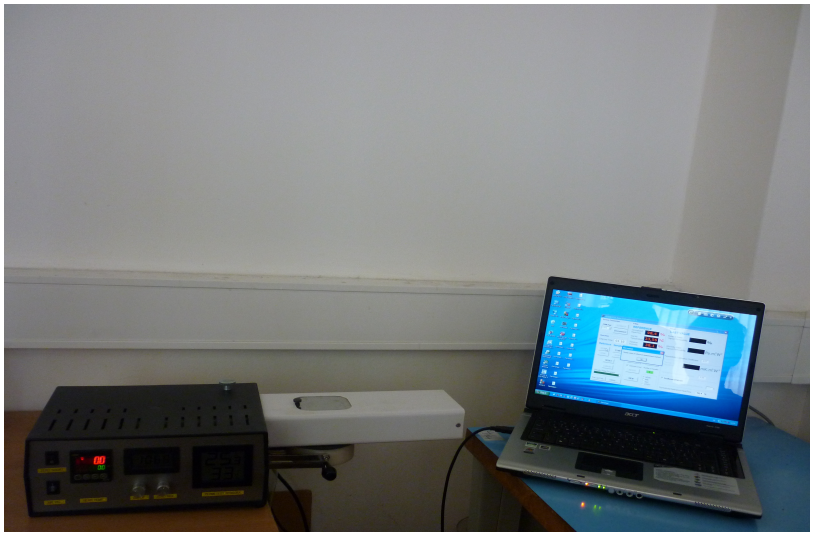


Fig. 4.6 Breathability machine

4.4.1 Measuring breathability and discussion of results

Breathability represents how much perspiration vapor can escape through a fabric from the inside out. Breathability depends on coating thickness, more coating the more waterproof of material causing the material to be less breathable. In fig.4.7, a 100% polyester twill weave coated twice with Texaflam XPE ECO (SB/Br-free) coating gave results of 0.10g/m^2.Pa.h and also Texaflam XPE ECO (SB/Br-free) coating that was applied once gave results of 0.15g/m^2.Pa.h. These results show less breathability comparing to the original material. Breathable allows or have ability to block out rain and snow while allowing vapour from sweat to evaporate. Polyester absorb little water and good at wicking perspiration. Coated fabrics are considered to be more uncomfortable to wear than water-repellent fabric as they are relatively stiff. Foam coating with Texaflam XPE (SB/Br) increase the pores, making the 100% polyester to be more breathable. Both 250gl^{-1} and 500gl^{-1}Texaflam XPE (SB/Br) gave 0.22g/m^2.Pa.h. Impregnation with Texaflam PE concentrated gave 100% polyester twill weave breathability of 0.23g/m^{2}Pa.h and also 500gl-1 Texaflam BS breathability was 0.28g/m^{2}Pa.h. Foam and impregnation of 100% polyester twill weave opened the pores.

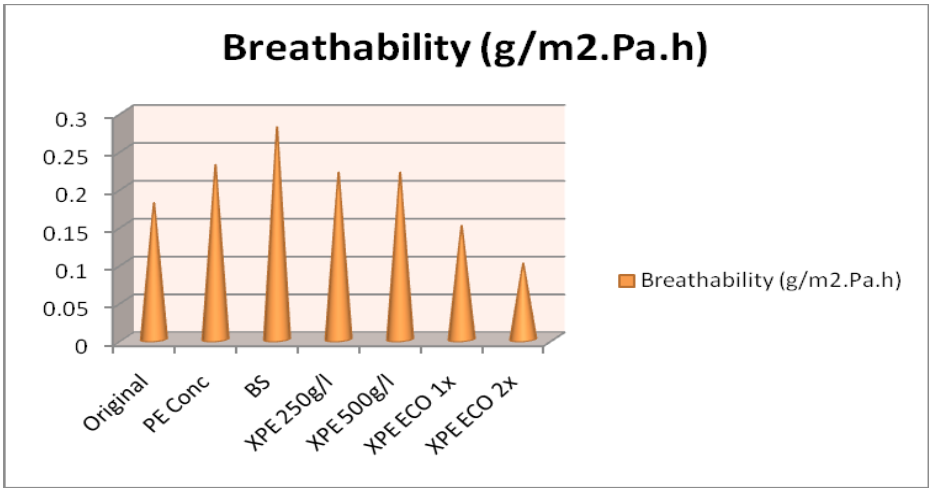


Fig. 4.7 Breathability values of 100% polyester twill weave

In fig. 4.8 original 65% polyester / 35% cotton twill weave gave breathability value of 0.23g/m^{2}Pa.h. Impregnation and coating cause the breathability to decreased. But impregnation with Texaflam PE concentrated did not change breathability, it remained 0.23g/m^{2}Pa.h. 65% polyester / 35% cotton twill weave coated once with Texaflam XPE ECO (SB/Br-free) was less breathable giving 0.09g/m^{2}Pa.h.Other drastically changes were of the use of 500gl^{-1}Texaflam

XPE (SB/Br) that gave breathability of 0.14g/m^{2}Pa.h. In the use of blends, it can be seen that the values of breathability decreases even after coating unlike in 100% polyester twill weave.

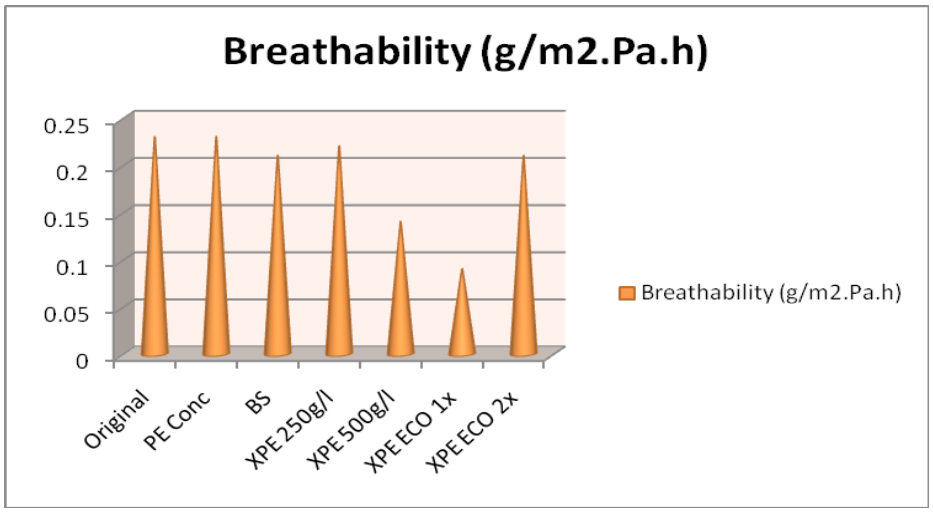


Fig. 4.8 Breathability values of 65% polyester/35% cotton twill weave

5. Color Difference and strength

5.1 Measuring of color difference and its results

Color difference, n – color difference calculated by using the CIE (International Commission on Illumination). There are three ways to calculate color difference, three formulas the CIE refined during years CIE76, CIE94, CIEDE 2000. Studying of depth color difference or even small color difference is critical. CIEDE 2000 is the most advanced way to determine color difference. There is norm color system X, Y, Z and it has superset of RGB. X, Y, Z are calculated through color matching function and are all positive Y represents the brightness and (x, y) represents saturation and hue. CIE Lab is a color scale based on opponent color theory. L* a* b* opponent-color scales (also referred to as CIELAB), based on applying a cube-root transformation to CIE 1931 tristimulus values X, Y, Z or CIE 1964 tristimulus values X10, Y10, Z10.

Tristimulus values when encoding. X, Y and Z are all calculated through color-matching functions and are always positive “L” represents light dark, an “a” represents red-green and “b” represents yellow-blue. RGB can be converted to XYZ having RGB values (Red, Green and Blue) as base 10 integers from 0 to 255. Two other color difference equations are L c h or CMC. “L” represents lightness to darkness (+L or –L); C represents Chroma from neutral to fully saturated color (+C,-C) and h is hue. Another color difference is delta e and is a total color difference. [42]

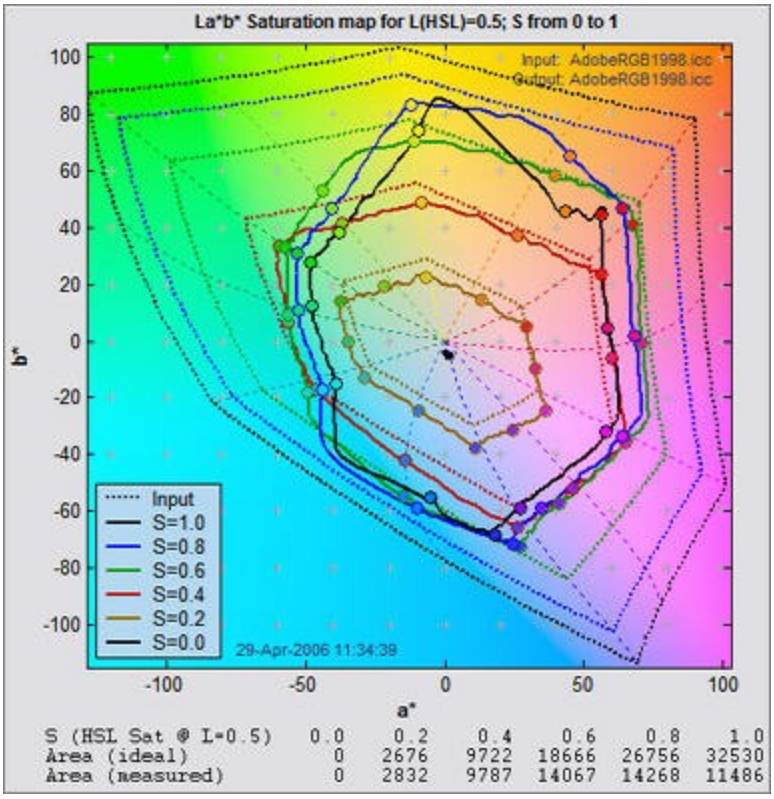


Fig 5.1 2D a* b* saturation map

Illuminant/Observer: D65 10 Deg

Standard Coordinates: L* = 58.66 a* = 6.37 b* = 17.16 C* = 18.30 h = 69.63

Table 5.1 Color difference and strength of 100% polyester twill weave

Sample No	DL*	Da*	Db*	DC*	DH*	DE*	Strength %
PE Conc.	-3.61	2.42	-1.78	-0.59	-2.94	4.69	122.12
BS	-0.17	0.52	0.23	0.40	-0.40	0.59	101.63
XPE 250 g/l	-1.18	0.48	0.58	0.71	-0.24	1.40	109.76

XPE 500 g/l	-0.18	0.22	0.26	0.32	-0.11	0.38	101.81
XPE ECO 1x	-0.22	0.40	0.54	0.64	-0.19	0.71	102.79
XPE ECO 2x	0.32	-0.15	-0.16	-0.20	0.09	0.39	97.54

Illuminant/Observer: D65 10 Deg

Standard Coordinates: L* = 76.39 a* = 3.99 b* = 14.74 C* = 15.27 h = 74.84

Table 5.2 Color difference and strength of 65% polyester/ 35% cotton twill weave

Sample No	DL*	Da*	Db*	DC*	DH*	DE*	Strength %
PE Conc.	-0.18	-0.03	-0.46	-0.45	-0.09	0.49	100.39
BS	-1.10	0.12	0.56	0.57	0.03	1.24	111.76
XPE 250 g/l	-0.21	0.10	0.65	0.65	0.08	0.69	103.68
XPE 500 g/l	-0.26	-0.08	-0.10	-0.12	0.05	0.29	102.07
XPE ECO 1x	0.10	-0.24	-0.57	-0.61	0.08	0.62	97.69
XPE ECO 2x	-3.08	2.18	0.49	1.16	-1.90	3.80	130.68

5.1.1 Discussion of color difference of samples

In table 5.1, after impregnation by 200gl^{-1} Texaflam PE concentrated of 100% polyester twill weave, decrease in lightness and become darker and CIEDE color difference of 4.69. The original polyester twill weave lightness was 58.66 and chroma was 15.27. Sample impregnated with 500gl^{-1} Texaflam BS was more saturated and CIEDE color difference of 0.59. Foam coating with 250gl^{-1} Texaflam XPE coating, sample was darker and more saturated with CIEDE color

difference of 1.40. Polyester twill weave coated twice with Texaflam XPE ECO was darker and more saturated. Texaflam XPE ECO coated once was darker and more saturated. Coating twice with Texaflam XPE ECO was less saturated.

In table 5.2, sample was less saturated and gave overall CIEDE color difference of 0.49. Impregnation with $500gl^{-1}$ Texaflam BS was darker and more saturated. Foam coating with $250gl^{-1}$ Texaflam XPE, sample was more saturated whereas foam coating with $500gl^{-1}$ Texaflam XPE had negligibly results no much difference to original sample. Coated twice with Texaflam XPE ECO coated twice was darker and more saturated. Texaflam XPE ECO coated once was less saturated. Coating twice with Texaflam XPE ECO was darker; more saturated and was redder with CIEDE color difference of 3.80.

5.2 Strength of textiles used

Strength is the level of resistance exhibited by a material against separation into individual pieces. In textile testing, strength usually mean tensile strength (breaking strength, limit strength, tear strength). Depending on the type of separation process, there are different types of strength, such a bursting strength; Flexural strength or resistance; Tear strength; Resistance to tear propagation; Crease resistance; Flexing abrasion resistance; Loop strength, Torsional strength; Wet strength. The different methods of tear test procedure are reflected in different standards, which are characterized by different methods of sample preparation, their shape and size, the way of clamping and the length of torn fabric distance, as well as the way of reading and calculating the tear force. [26]

5.2.1 Measuring of strength and discussion

The tensile strength of the samples used were evaluated according to the ASTM D5587 -

08 standard. Rectangular specimens 30 cm long, and 5 cm width were tested under a constant speed. The specimens were each clamped in a vertical position between parallel jaws, one stationary and one movable.

Impregnation with 200gl^{-1} Texaflam PE concentrated show more strength than all samples tested for strength in table 5.1, with strength of 122.12%, followed by sample which was coated by 250gl^{-1} Texaflam XPE foam coating with strength of 109.76. Impregnation with 500gl^{-1} Texaflam BS and 500gl^{-1} Texaflam XPE gave strength of approximately 101. The samples which had the least strength was coated samples with Texaflam XPE ECO coated once which gave 102.79 and coated twice Texaflam XPE ECO was 97.54. Showing that 100% polyester twill weave after coating lost its strength.

In table 5.2, coated sample twice with Texaflam XPE ECO show strength of 130.68%, followed by sample which was impregnated with 500gl^{-1} Texaflam BS, with strength of 111.76%. Coated sample with 250gl^{-1} Texaflam XPE foam coating had strength of 103.68 and 102.07% foam coating with 500gl^{-1} Texaflam XPE. Impregnation with 200gl^{-1} Texaflam PE concentrated was 100.39 and lastly sample coated once with Texaflam XPE ECO was 97.69. Coating twice 65% polyester/35% cotton twill weave with Texaflam XPE ECO made it to be much stronger.

6. Differential scanning calorimetry

6.1 Detection of phase transitions

Differential scanning calorimetry measures heat and by doing so heat is exchanged. The samples are tested in a relation to their physical or chemical changes during a continuous, linear sample warming or cooling. Temperature change is effected by heat exchange in a body that can

be used as a measure of the heat exchanged. Differential Scanning .DSC's are used to measure heat flow rates and characteristics temperatures of a reaction or a transition as well.
The measurement of heat already have integral (total) heats of reaction or transition and also the determination of partial heats developed within a selected temperature interval. For kinetic evaluations, determination of crystallinity and purity such values are of importance.

Differential scanning calorimetry or DSC is a thermo-analytical technique in which the differerences in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. DSC measures the change of a property for instance heat flow rate difference. Heat flow rate difference is normally released due to an alteration of the sample temperature; there is no change of heat flow rate when there is no alteration of the sample temperature difference can be measured. Heat flows only if there is a temperature difference present meaning a non-zero heat flow rate difference implies a temperature difference between the sample and the reference and their surroundings and a change of heat flow rate difference implies a change of temperature. The main application of DSC is in studying phase transitions, such as melting, glass transitions, or exothermic decompositions. These transitions involve energy changes or heat capacity changes that can be detected by DSC with great sensitivity. [39]

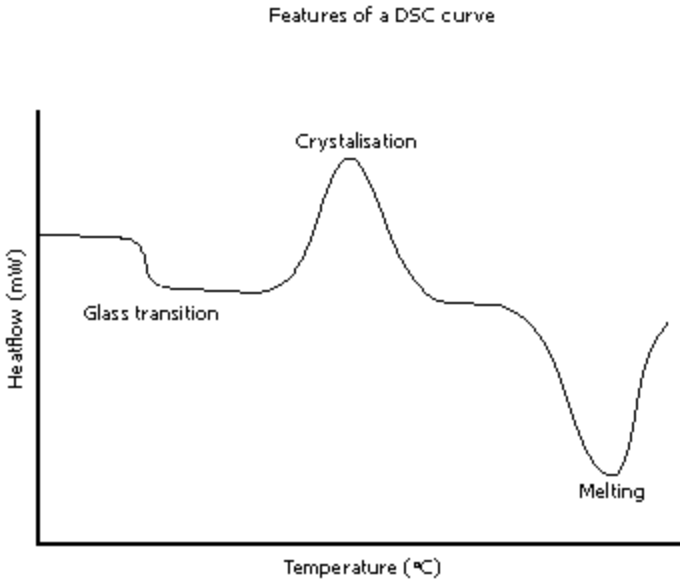


Fig. 6.1 A Schematic DSC curve [40]

Thermal decomposition of cotton produces solid residues, liquid materials and volatile gases when it is heated without oxygen. Pyrolysis of cellulose fiber includes three stages: initial, main, and char decomposition. The main pyrolysis stage of cotton occurs in the temperature range of 300–380 °C. In this stage, the weight loss is very fast and significant. The char pyrolysis occurs at the temperature above 430 °C. Chemical modification of fibers usually occurs at their amorphous regions, where the flame-retardants are chemically incorporated onto cotton. During cotton pyrolysis, flame-retardants catalyze reactions of dewatering, decarboxylation and charring.

Semi crystalline thermoplastics and their physical properties depend on molecular structures, crystalline and amorphous structures. The crystalline regions impart mechanical strength and amorphous region is flexible Crystallinity in a polymer on different polymer molecules, molecular weight, cross linking, the steric orientation, concentration of additives, thermal and mechanical history of polymer formulation. In differential scanning calorimetry, the change in heat capacity is shown by a curve whereas glass and melt transition is display as function of temperature. [41]

6.2 Measuring DSC values

The materials were studied under heat using differential scanning calorimetry. Measurement of the DSC values was done on the DSC 6 instrument. The differential scanning calorimetry (DSC) is a calorimetric method observing temperature effects of the researched sample connected to its physical or chemical changes during gradual linear warming or cooling. Also to observe thermal transition or changes taking place when the materials were heated. Sample was put in small capsule or sample pan.

Optimum number of sample was 10mg and capsule alone was 24.5 mg when weighed. The machine had reference pan and computer is monitoring temperature and regulate heat flow. Each capsule or pan sits on top of a heater. The reference pan is empty and it is important to heat both pans at the same rate. As a result sample pan takes more heat to keep the temperature at the

same level with reference pan. Initial warming was up to 25°C and remaining at that temperature for 1 minute then warming from 25°C to 280°C at steps 15°C per 1 minute. Whereas cooling was from the temperature of 280°C to 20°C, in steps at 15°C per 1 minute. The measured data was recorded by the software (PE Pyris Series – DSC 6) installed on the connected computer; the DSC curves are plotted on fig. 6.3 up to 6.16.

Fig. 6.2 Differential Scanning Calorimetry

6.3 Results and discussion of DSC

In the graphs below shows heat and temperature intervals of the melting point and recrystallization that has been received and released. The derivative of each graph can also be observed from each graph. In all polyester/cotton sample a slightly curve is observed at 40°C to 120°C temperatures where cotton is slightly swelling. The x-axis shows the temperature [◦C] and the y-axis shows the heat flow [mW]. The pyrolysis of cotton includes three stages: initial, main, and char decomposition. The related temperature, speed and weight loss of every stage can be found from the TGA curve. In the initial stage, where the temperature range in this case is below 280 °C, the most important changes of the fibers are some physical properties and little weight loss. Here, the damage of the cotton occurs mostly in amorphous region of the polymers. The

main pyrolysis stage occurs in the temperature range of 300 – 380 °C. In this stage, the weight loss is very fast and significant.

The char pyrolysis occurs at the temperature above 430 °C. The pyrolysis decomposition temperatures and weight loss of the flame-retardant cotton shows is lower than that of the untreated cotton. It is found that the decomposition temperature of the second stage is about 30 °C lower than that of the untreated cotton, indicating the effluence of flame-retardants. The solid residue resulting from the flame-retardant cotton is significantly higher, showing that more charred products produced after the flame-retardant treatment. At the same time in this case cotton was blended with polyester when cotton slightly swells, polyester melts on cotton forming a solid char. It is possible to observe that there are no much differences between the enthalpy of the main peaks of the different substrates.

Fig. 6.3 shows the DSC curve for the 100% polyester used in this diploma thesis. Melting temperature ranges in the interval 238 – 262°C, value of the "peak" is approx. 255°C. The heat received by the sample is 59.802 Jg^{-1}. The temperature of recrystallization lies in the interval of 214 – 175 °C; the "peak" is approximately 194°C. Heat released by the sample is -45.622 Jg^{-1}.

Fig. 6.4 shows the DSC curve for 100% polyester impregnated with 200g/l Texaflam concentrated used in this diploma thesis. Melting temperature ranges in the interval 225 – 255°C, value of the "peak" is approx. 246°C. The heat received by the sample is 50.648 Jg^{-1}. The temperature of recrystallization lies in the interval of 211 – 181 °C; the "peak" is approximately 197°C. Heat released by the sample is -51.123 Jg^{-1}.

Fig. 6.5 shows the DSC curve for 100% polyester impregnated with 500g/l Texaflam BS used. Melting temperature ranges in the interval 245 – 262°C, value of the "peak" is approx. 255°C. The heat received by the sample is 44.328 Jg^{-1}. The temperature of recrystallization lies in the interval of 229 – 209 °C; the "peak" is approximately 222°C. Heat released by the sample is -36.406 Jg^{-1}.

Fig. 6.6 shows the DSC curve for 100% polyester coated with 250g/l Texaflam XPE (SB/Br) foam coating used. Melting temperature ranges in the interval 230 – 262°C, value of the "peak" is approx. 254°C. The heat received by the sample is 51.525 Jg^{-1}. The temperature of recrystallization lies in the interval of 220 – 192 °C; the "peak" is approximately 211°C. Heat released by the sample is -49.858 Jg^{-1}.

Fig. 6.7 shows the DSC curve for 100% polyester coated with 500g/l Texaflam XPE (SB/Br) foam coating used. Melting temperature ranges in the interval 230 – 266°C, value of the "peak" is approx. 254°C. The heat received by the sample is 51.491 Jg^{-1}. The temperature of recrystallization lies in the interval of 225 – 183 °C; the "peak" is approximately 209°C. Heat released by the sample is -51.990 Jg^{-1}.

Fig. 6.8 shows the DSC curve for 100% polyester coated with Texaflam XPE (SB/Br) coating 1x used. Melting temperature ranges in the interval 241 – 261°C, value of the "peak" is approx. 255°C. The heat received by the sample is 37.188 Jg^{-1}. The temperature of recrystallization lies in the interval of 216 – 180 °C; the "peak" is approximately 199°C. Heat released by the sample is -38.595 Jg^{-1}.

Fig. 6.9 shows the DSC curve for 100% polyester coated with Texaflam XPE (SB/Br) coating 2 xs used. Melting temperature ranges in the interval 241 – 261°C, value of the "peak" is approx. 254°C. The heat received by the sample is 28.260 Jg^{-1}. The temperature of recrystallization lies in the interval of 215 – 182 °C; the "peak" is approximately 204°C. Heat released by the sample is -29.117 Jg^{-1}.

Fig. 6.10 shows the DSC curve 65% PES /35% CO original used in this diploma thesis. Melting temperature ranges in the interval 242 – 260°C, value of the "peak" is approx. 255°C. The heat received by the sample is 36.849 Jg^{-1}. The temperature of recrystallization lies in the interval of 215 – 181 °C; the "peak" is approximately 200°C. Heat released by the sample is -28.153 Jg^{-1}.

Fig. 6.11 shows the DSC curve for 65% PES /35% CO impregnated with 200g/l Texaflam concentrated used. Melting temperature ranges in the interval 227 – 260°C, value of the "peak" is approx. 250°C. The heat received by the sample is 35.881 Jg^{-1}. The temperature of recrystallization lies in the interval of 217 – 192 °C; the "peak" is approximately 208°C. Heat released by the sample is -30.947 Jg^{-1}.

Fig. 6.12 shows the DSC curve for 65% PES /35% CO impregnated with 500g/l Texaflam BS used. Melting temperature ranges in the interval 243 – 261°C, value of the "peak" is approx. 254°C. The heat received by the sample is 33.274 Jg^{-1}. The temperature of recrystallization lies in the interval of 223 – 198 °C; the "peak" is approximately 211°C. Heat released by the sample is -29.406 Jg^{-1}.

Fig. 6.13 shows the DSC curve for 65% PES /35% CO coated with 250g/l Texaflam XPE (SB/Br) foam coating used. Melting temperature ranges in the interval 237 – 260°C, value of the "peak" is approx. 255°C. The heat received by the sample is 36.247 Jg^{-1}. The temperature of recrystallization lies in the interval of 213 – 181 °C; the "peak" is approximately 197°C. Heat released by the sample is -24.842 Jg^{-1}.

Fig. 6.14 shows the DSC curve for 65% PES /35% CO coated with 500g/l Texaflam XPE (SB/Br) foam coating used. Melting temperature ranges in the interval 234 – 261°C, value of the "peak" is approx. 254°C. The heat received by the sample is 32.968 Jg^{-1}. The temperature of recrystallization lies in the interval of 220 – 192 °C; the "peak" is approximately 209°C. Heat released by the sample is -29.607 Jg^{-1}.

Fig. 6.15 shows the DSC curve for 65% PES /35% CO coated with Texaflam XPE (SB/Br) coating 1x used. Melting temperature ranges in the interval 240 – 262°C, value of the "peak" is approx. 255°C. The heat received by the sample is 37.224 Jg^{-1}. The temperature of recrystallization lies in the interval of 215 – 176 °C; the "peak" is approximately 209°C. Heat released by the sample is -29.939 Jg^{-1}.

Fig. 6.16 shows the DSC curve for 65% PES /35% CO coated with Texaflam XPE (SB/Br) coating 2 xs used. Melting temperature ranges in the interval 241 – 261°C, value of the "peak" is approx. 254°C. The heat received by the sample is 21.836 Jg^{-1}. The temperature of recrystallization lies in the interval of 218 – 190 °C; the "peak" is approximately 209°C. Heat released by the sample is -21.116 Jg^{-1}.

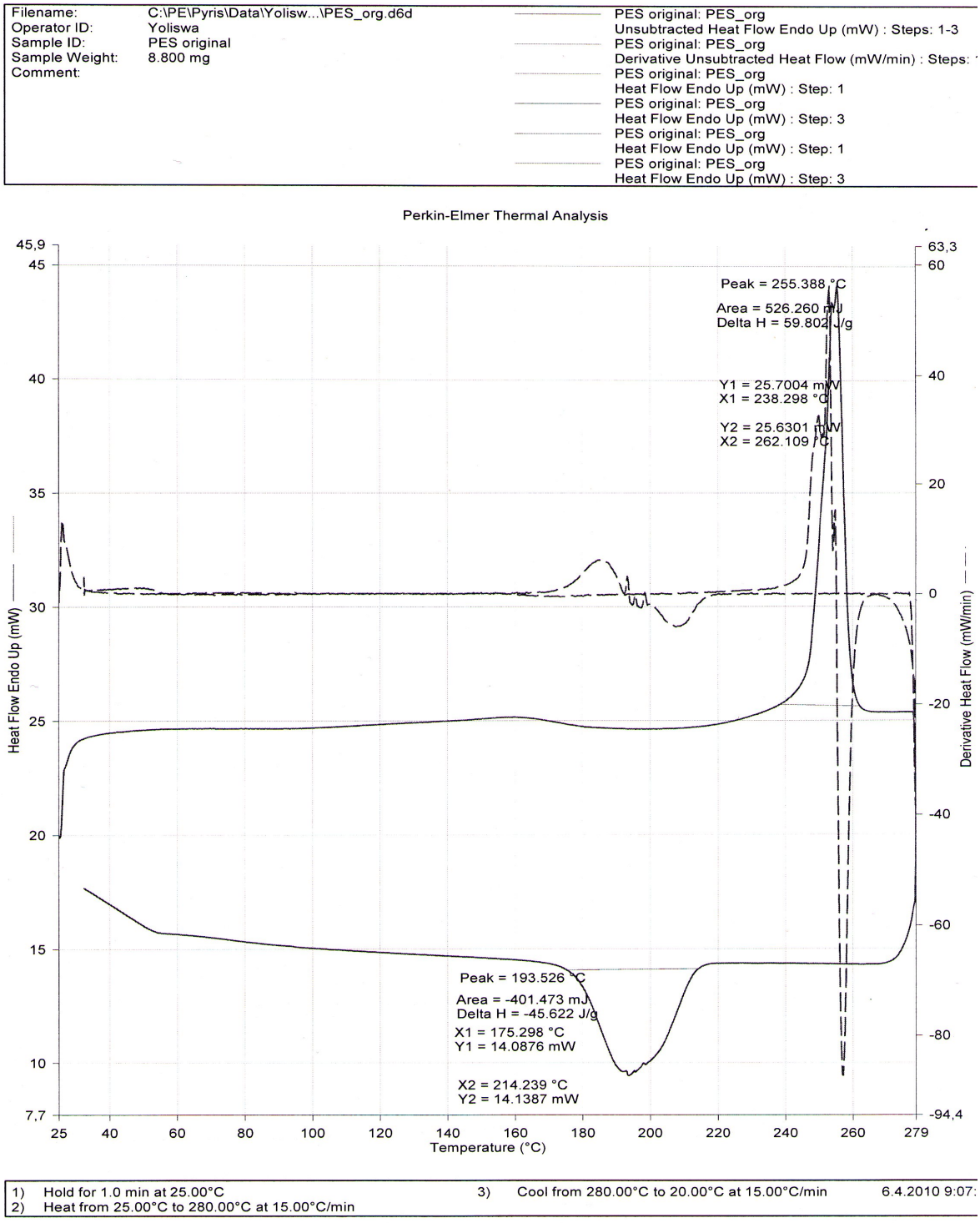


Fig. 6.3 DSC curve for 100% polyester original

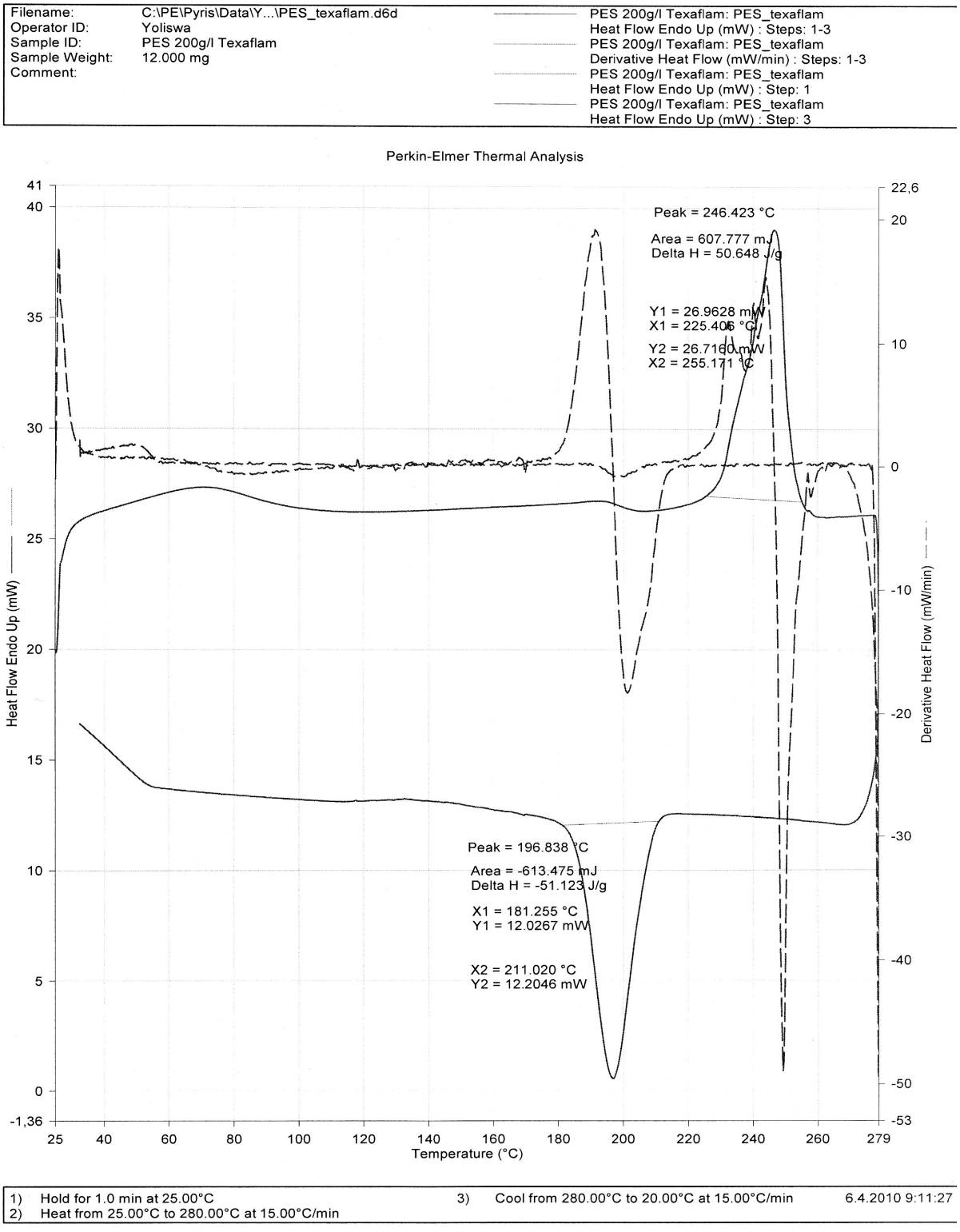


Fig. 6.4 DSC curve for 100% polyester impregnated with 200g/l Texaflam Concentrated

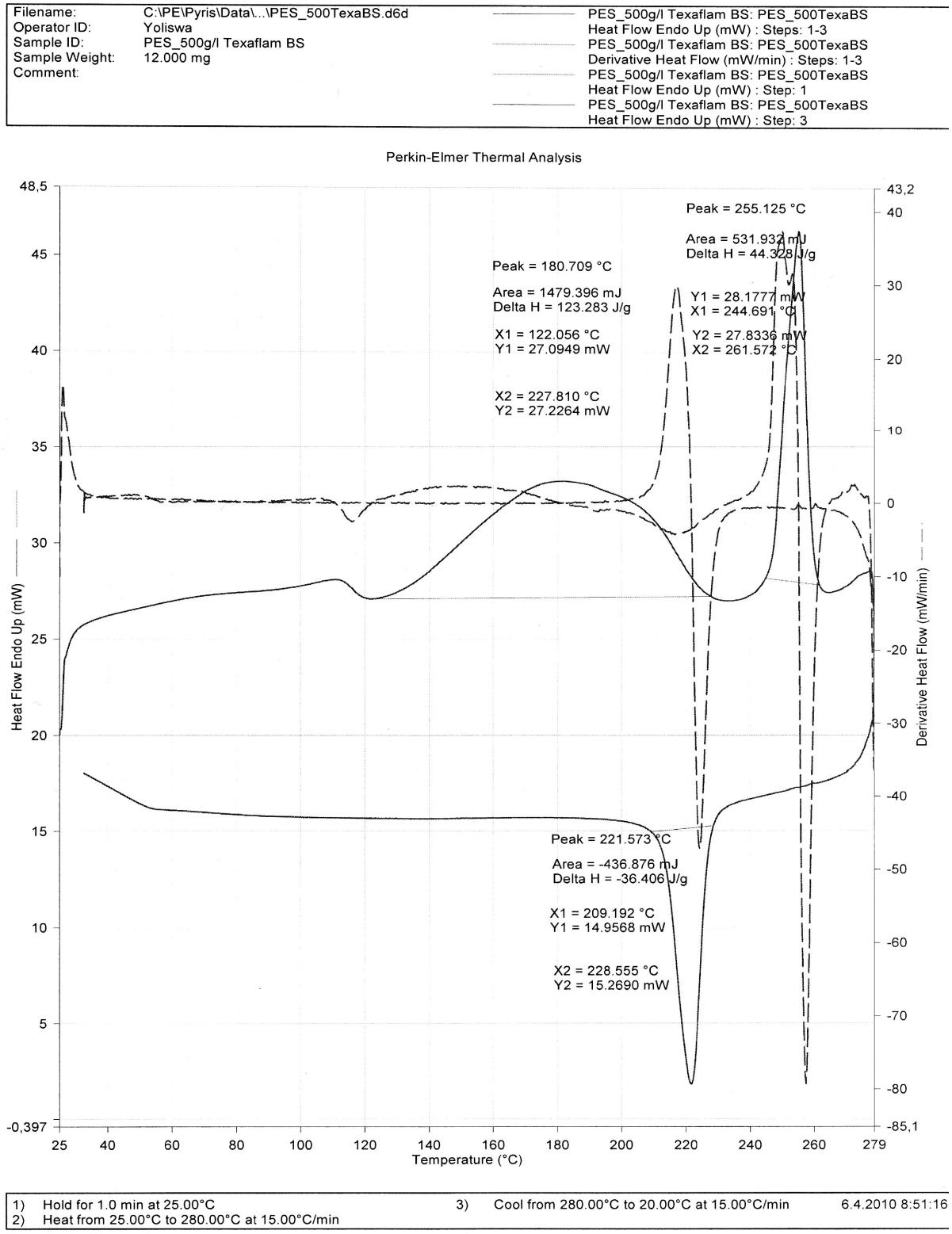


Fig. 6.5 DSC curve for 100% polyester impregnated with 500g/l Texaflam BS

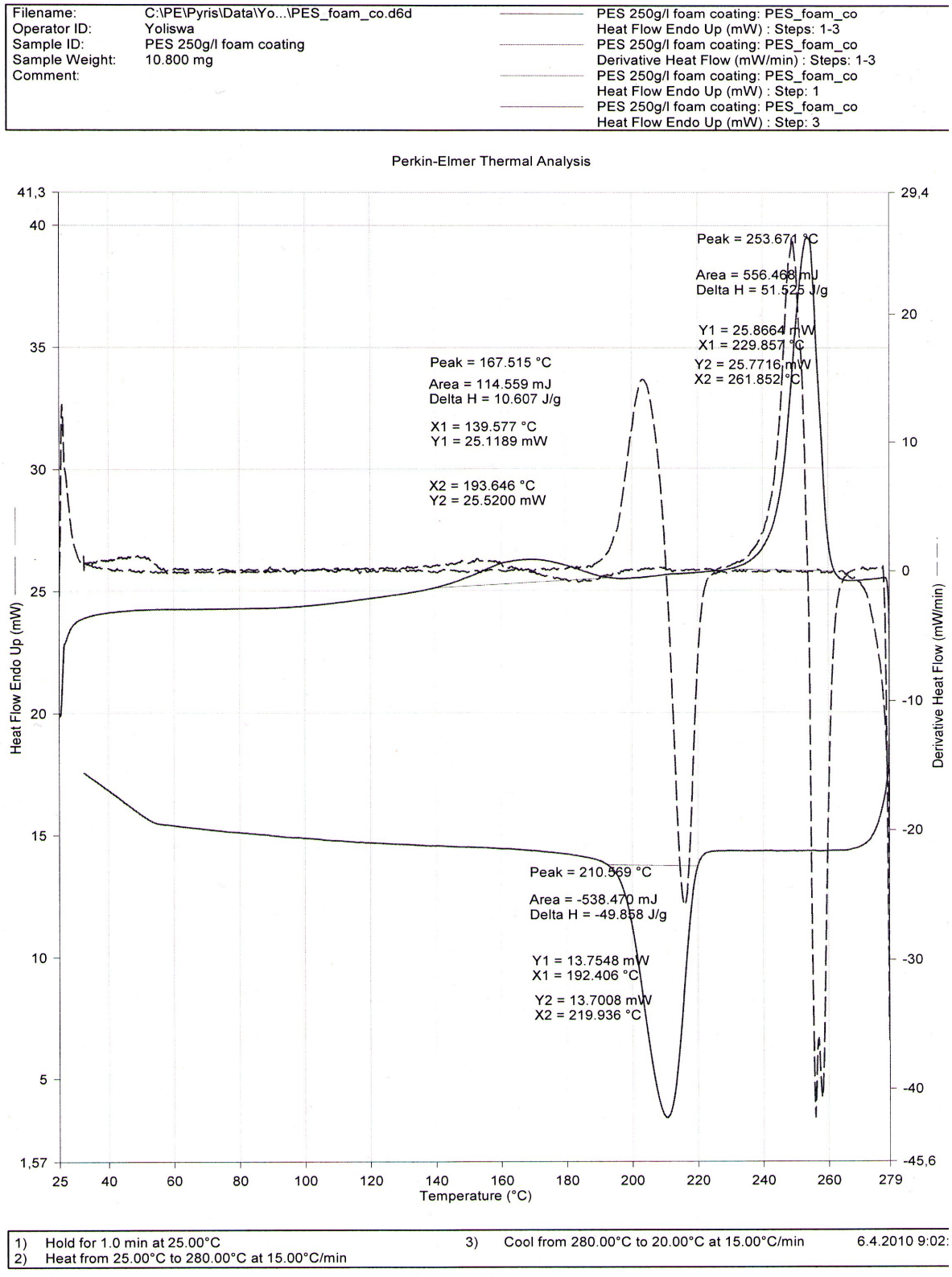


Fig. 6.6 DSC curve for 100% polyester coated with 250g/l Texaflam XPE (SB/Br) foam coating

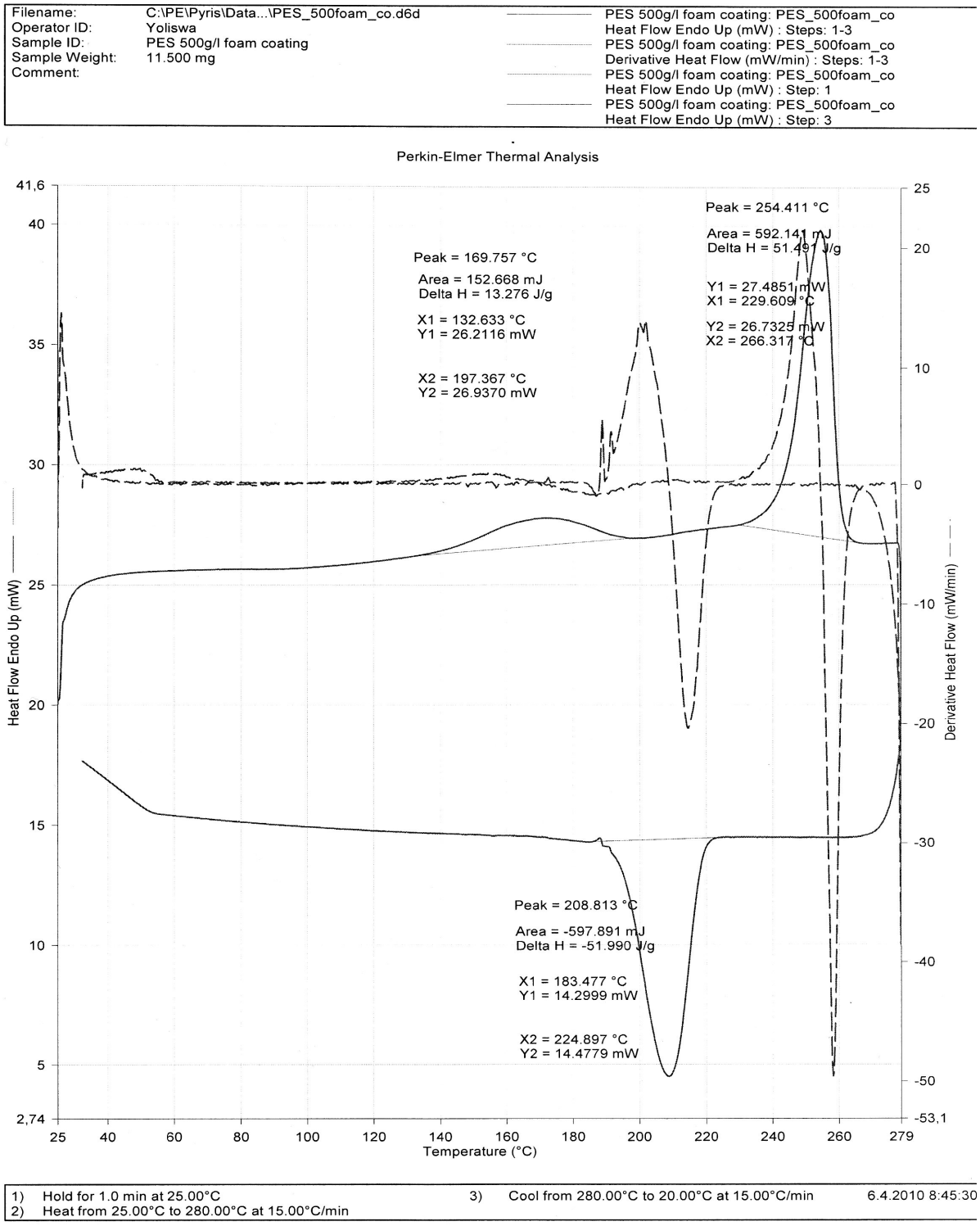


Fig. 6.7 DSC curve for 100% polyester coated with 500g/l Texaflam XPE (SB/Br) foam coating

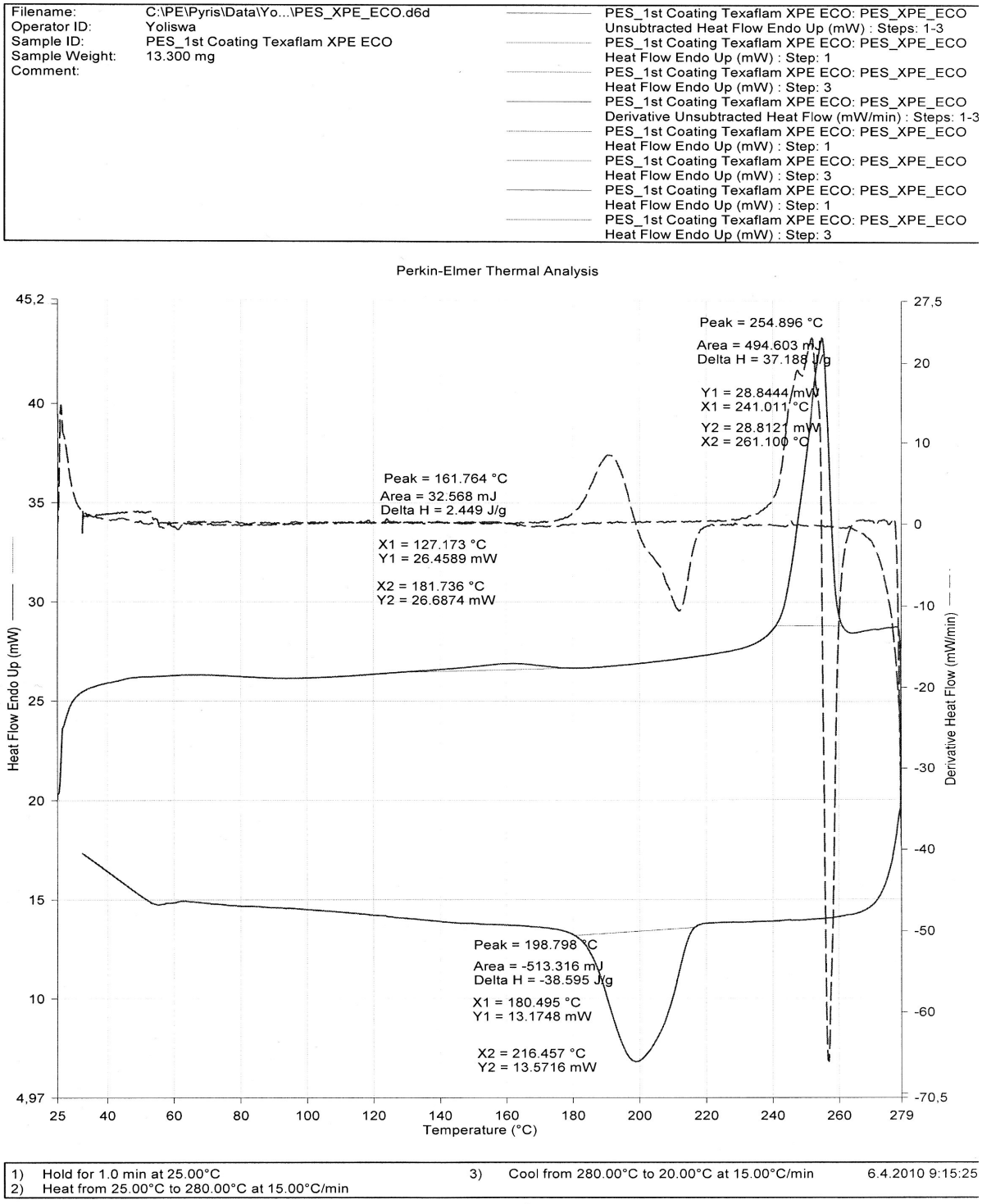


Fig. 6.8 DSC curve for 100% polyester coated with Texaflam XPE (SB/Br) coating 1x

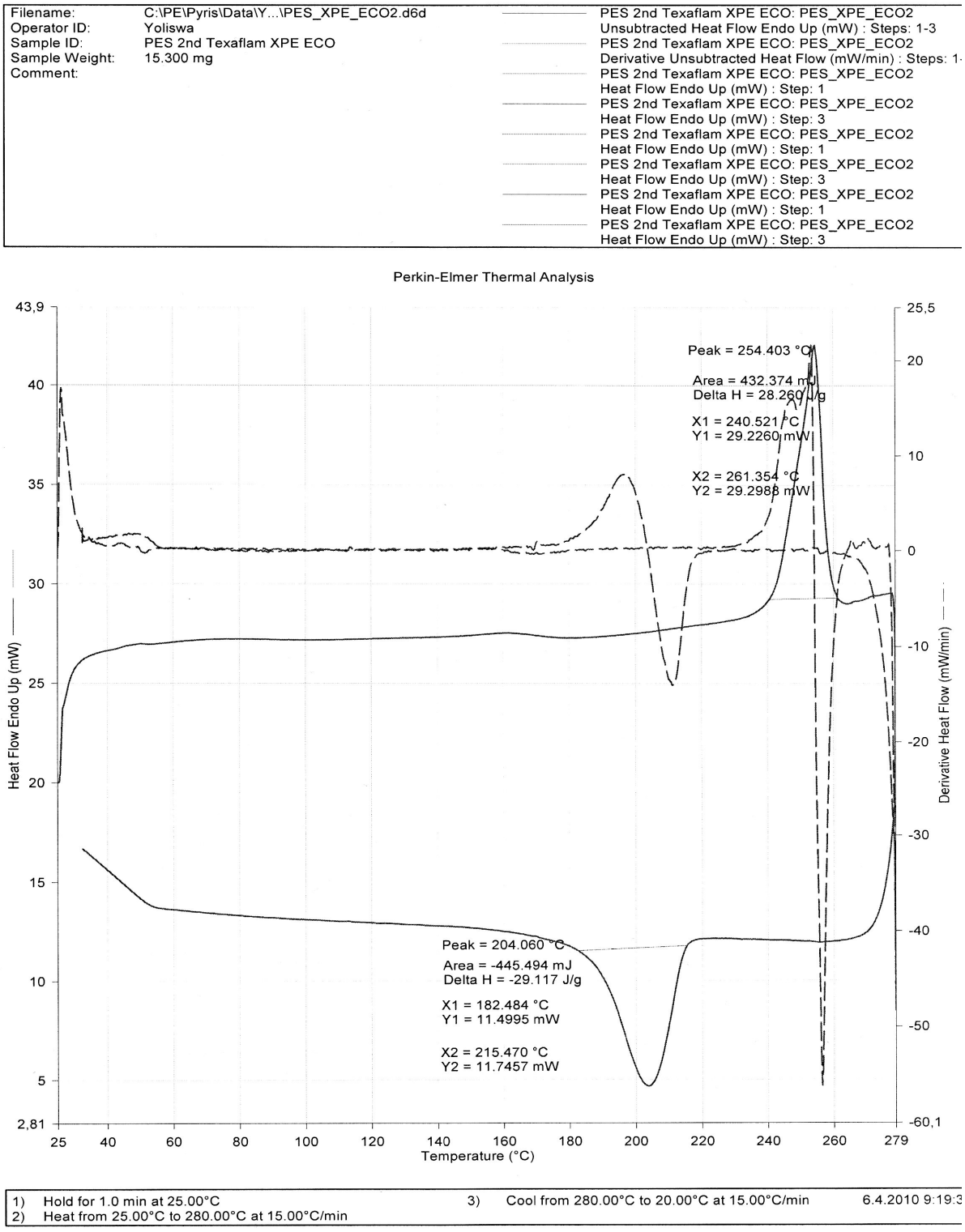


Fig. 6.9 DSC curve for 100% polyester coated with Texaflam XPE (SB/Br) coating 2x

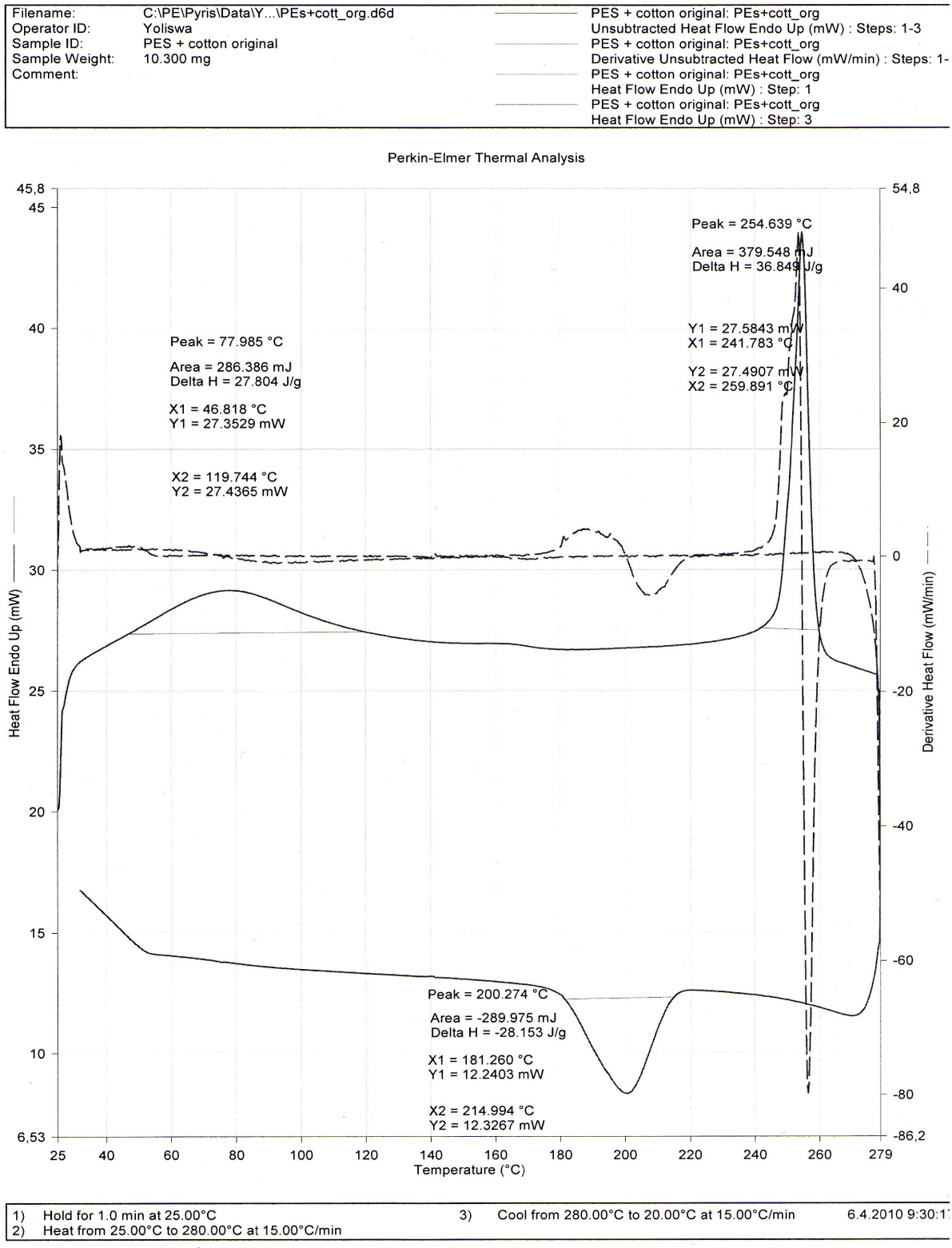


Fig. 6.10 DSC curve for 65% PES /35% CO original

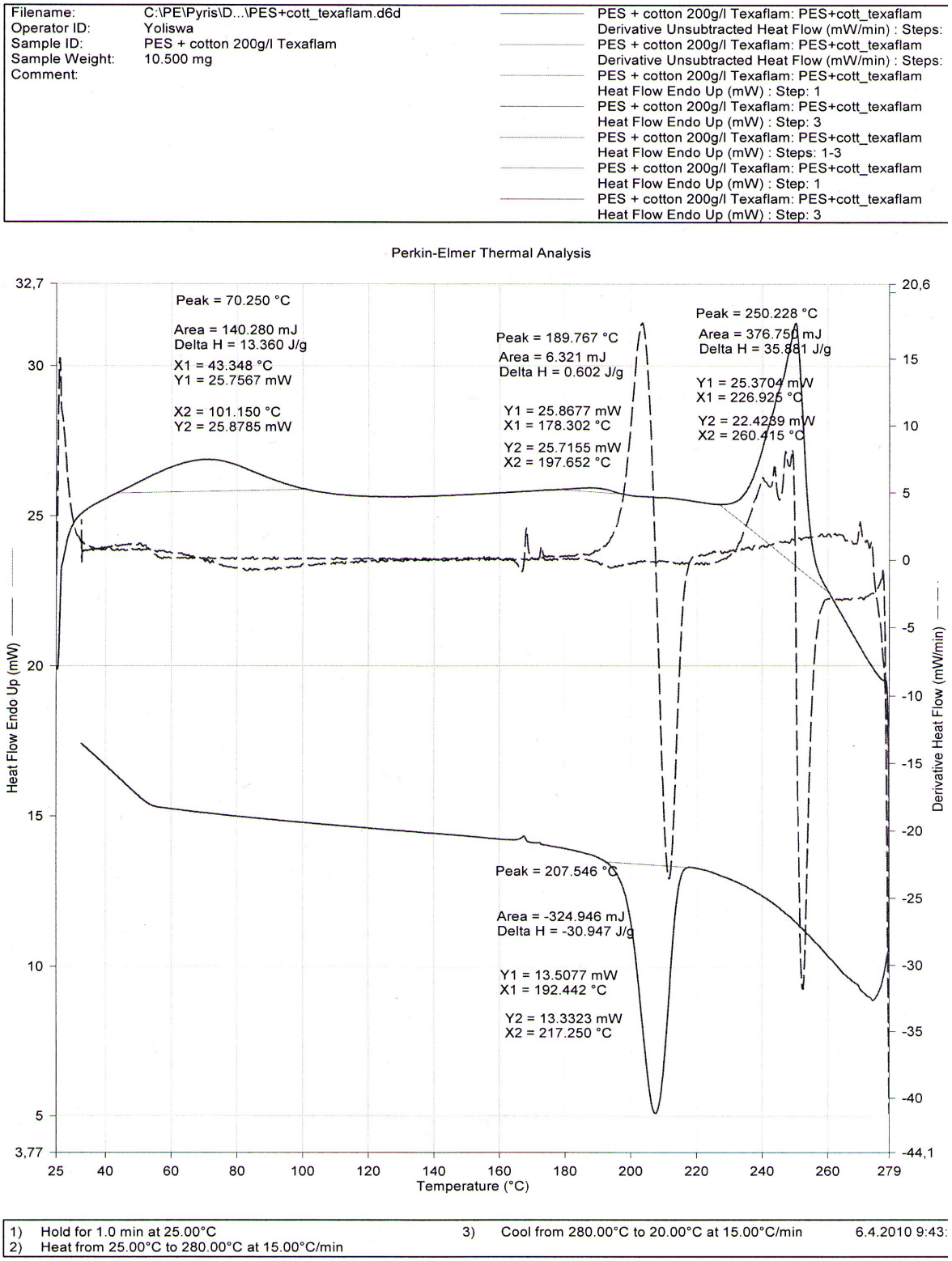


Fig. 6.11 DSC curve for 65% PES /35% CO impregnated with 200g/l Texaflam Concentrated

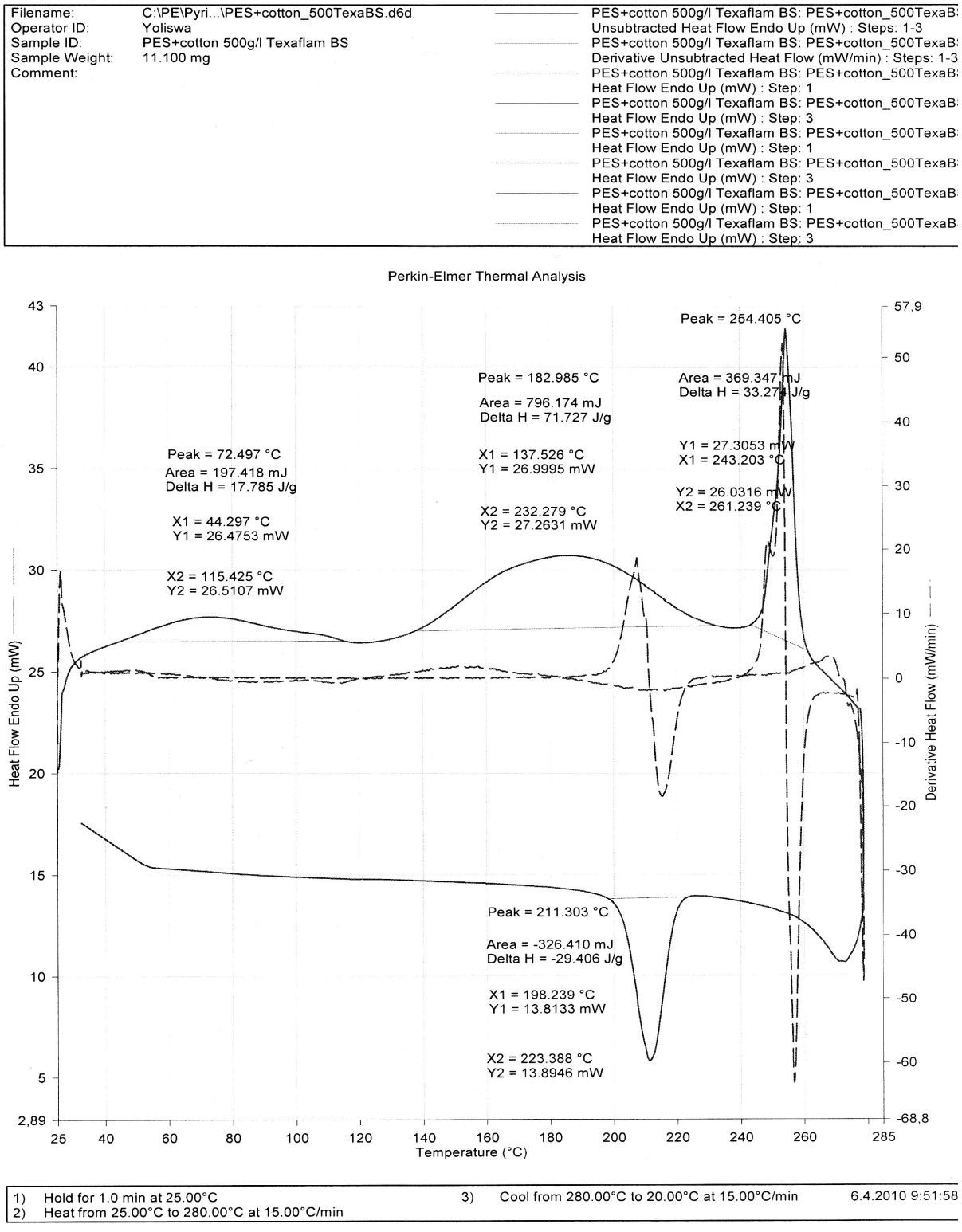


Fig. 6.12 DSC curve for 65% PES /35% CO impregnated with 500g/l Texaflam BS

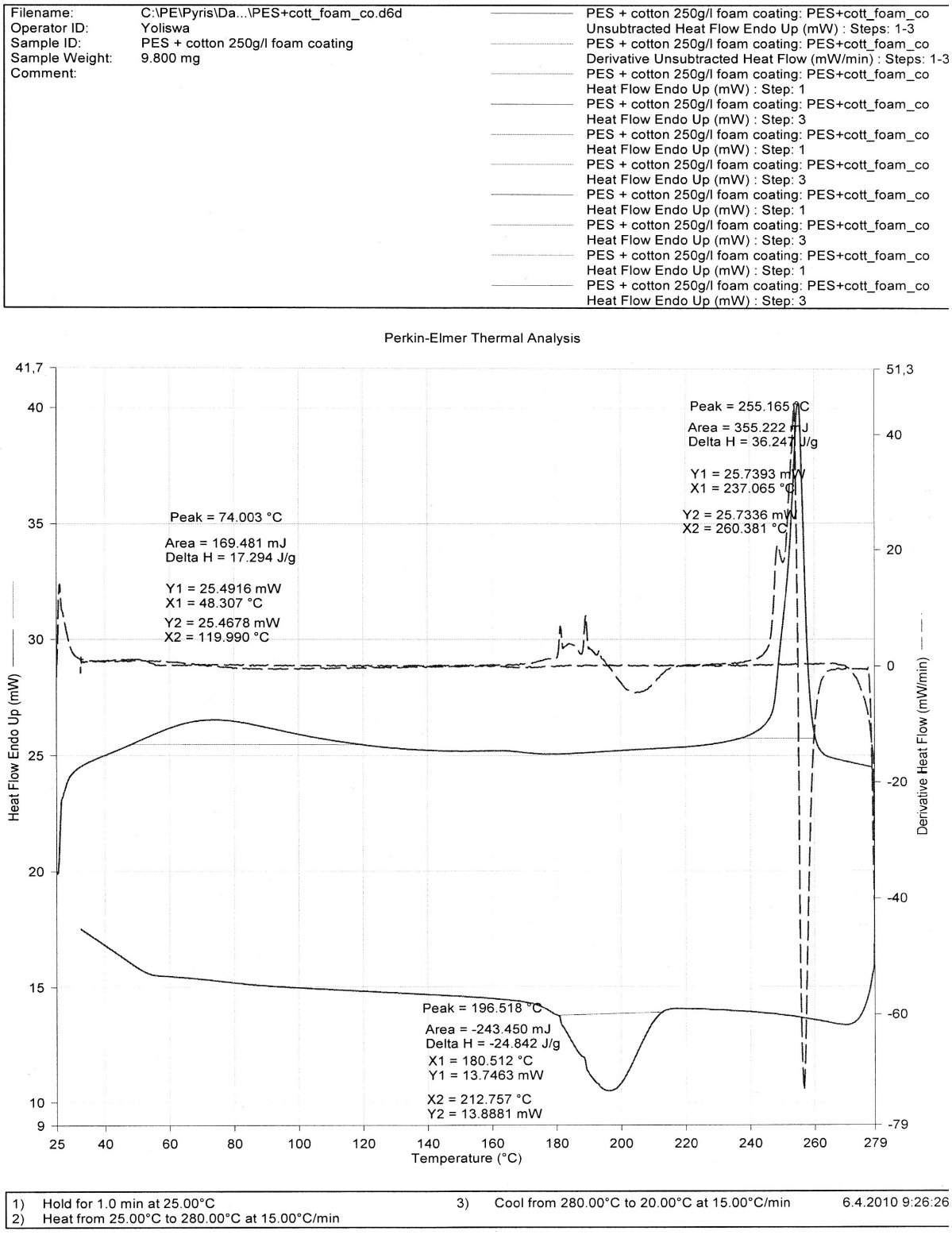


Fig. 6.13 DSC curve for 65% PES /35% CO coated with 250g/l Texaflam XPE (SB/Br) foam coating

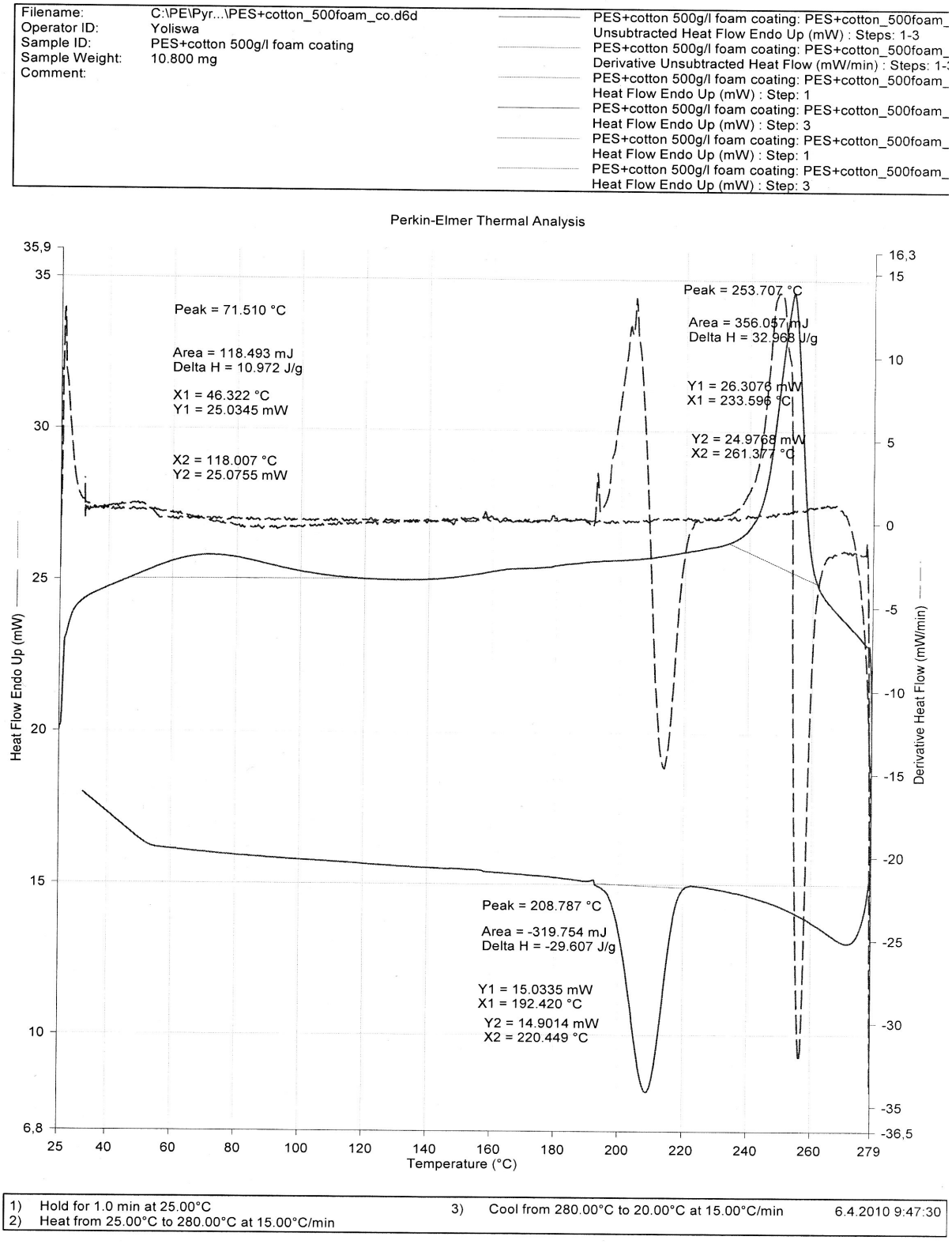


Fig. 6.14 DSC curve for 65% PES /35% CO coated with 500g/l Texaflam XPE (SB/Br) foam coating

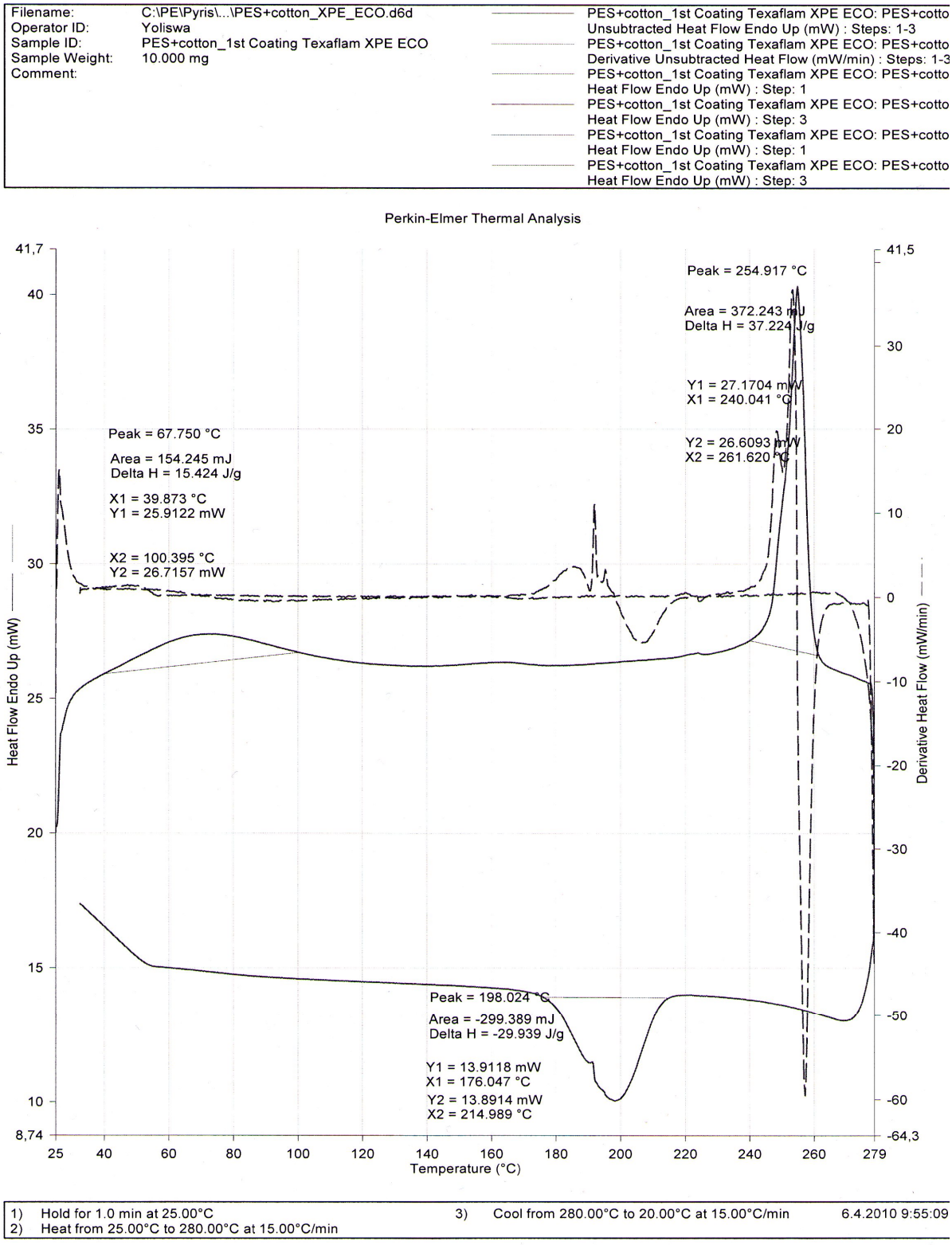


Fig. 6.15 DSC curve for 65% PES /35% CO coated with Texaflam XPE (SB/Br) coating 1x

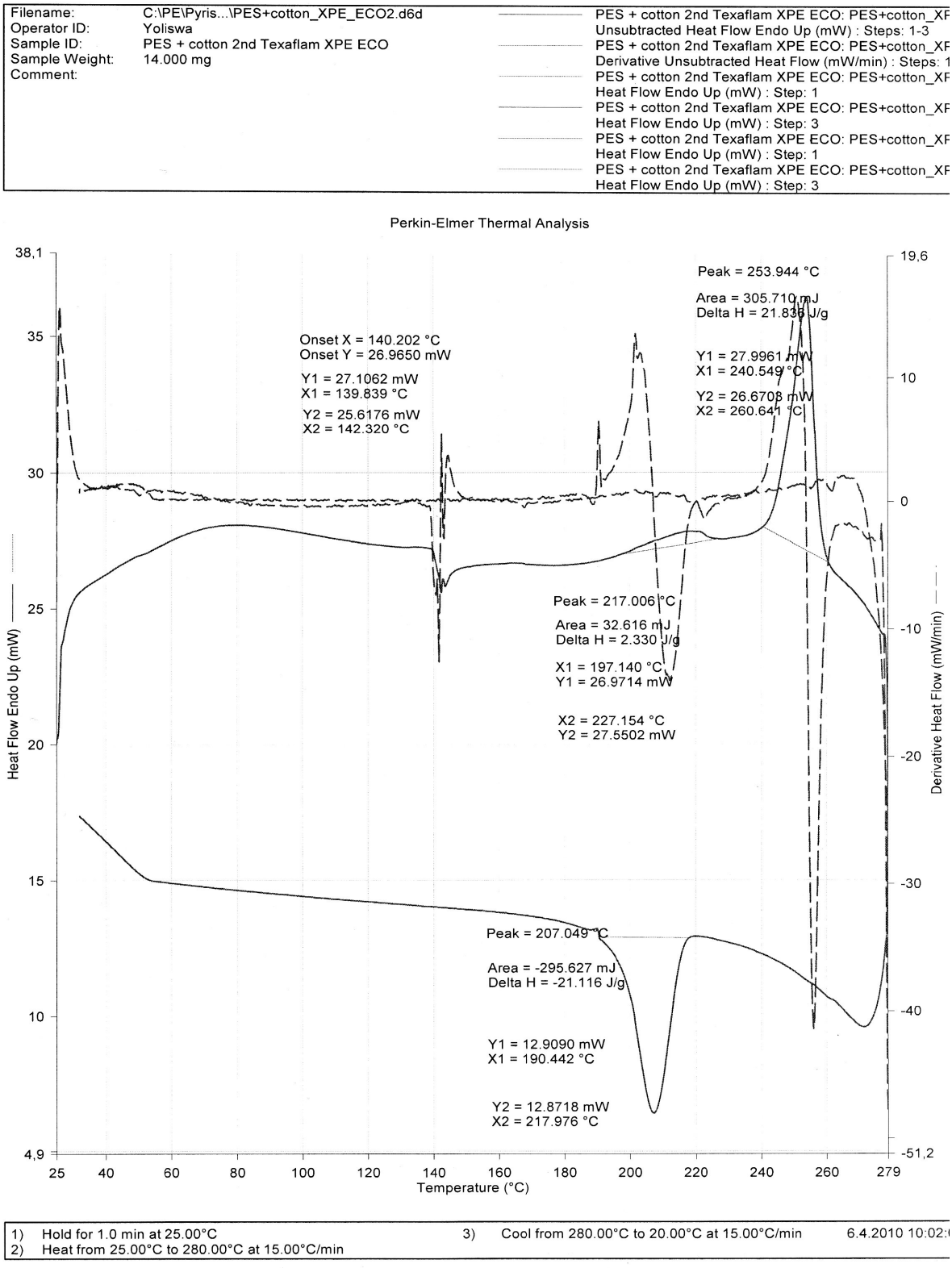


Fig. 6.16 DSC curve for 65% PES /35% CO coated with Texaflam XPE (SB/Br) coating 2 xs

7. Laser

7.1 Laser treatment and its device

Fields of laser applications are very wide. It can be used in scientific, military, medical and commercial applications. It could be used for also metals, plastics, glass, wood, leather and textiles etc.

The Marcatex 150 Flexi is a CO_2 laser system excited by high frequency, designed as a cutting, welding of engraving instrument for industrial applications.

This wavelength is invisible to the human eye and is in the infra-red part of the electromagnetic spectrum. The CO_2 laser radiation can be reflected by smooth metallic surfaces, even if they are darkened.

The influence of laser beam to textile is thermal. Laser beam causes decolouring, yellowing and sometimes pyrolysis of natural fibers. A different situation is occurred at synthetic fibers, which are melted.

Fig. 7.1 Laser device – Marcatex 150 Flexi

In textile industry laser surface designing is a modern way how to modify the surface of

textiles. The principle consists in the influence of laser beam to material.

General description:

Model	Marcatex 150 Flexi
Average power	150 watts
Peak power	230 watts
Working frequency	50/60 Hz
Wavelength of laser beam	10.6 μm
Polarization	linear

The samples were 100% polyester and blended 65% polyester/ 35% cotton twill weaves. Both originals were used. Also impregnated fabrics with 200gl^{-1} Texaflam PE, 500gl^{-1} Texaflam BS and some coated with 250gl-1 Texaflam XPE (SB/Br) foam coating, 500gl-1 Texaflam XPE (SB/Br) foam coating, Texaflam XPE ECO (SB/Br-free) coating 1x and Texaflam XPE ECO (SB/Br-free) coating 2xs. The specimens were cut 20cm x 5cm and put at 45^{o} angle. The wavelength of laser beam was directed to all the specimens used in this diploma thesis and power used was 100kW at different energies which was 5J and 10J. When energy is 5J the laser beam is much stronger than 10J.

Low frequency means a short pulse duration and thus high peak power, which presents that in a short time a high peak power, which presents that in short time a high energy will be applied to the object surfaces. If the deflection speed is too high, the laser points will be far apart that no closed lines are attained. But if the deflection speed is too low then the points will overlap too much and that is what is desired.

7.2 Results of laser treatment and discussion

Burning original samples with laser both 100% polyester twill weave and blended 65% polyester/ 35% cotton were completely burning without stopping. When radiation strikes the sample surface the energy was absorbed. The type of interaction of the beam with the surface depended on type of material and additive content, including the effect of surface coatings. At higher intensity, 65%PES/ 35%CO and 100% polyester twill weaves impregnated with 200g/l Texaflam PE concentrated approximately average 20mm. Also 65% PES/35% CO and 100% polyester twill weaves impregnated with 500g/l Texaflam BS were burned into an oval shape with a diameter of approximately average 19mm. Whereas at lower intensity, 100% polyester twill weave impregnated with 200g/l Texaflam PE concentrated and 100% polyester twill weave impregnated with 500g/l Texaflam BS had oval shape of 15mm after burning. Impregnated 65% PES/35% CO twill weave with 200g/l Texaflam PE and 65% PES/35% CO twill weave impregnated with 500g/l Texaflam BS at low intensity were partial burned at lower intensity with average diameter of 10mm.

At higher intensity, two samples of 100% polyester twill weave coated with 250g/l Texaflam XPE (SB/Br) foam coating was 30mm diameter and other with 500g/l Texaflam XPE (SB/Br) foam coating when radiation strikes the samples were burned into an oval shape with diameter approximately 28mm and both at lower intensity the diameter was approximately 20mm. Showing that using laser on Texaflam XPE foam samples gives worse results because 65% PES /35% CO twill weave coated with 250g/l and 500g/l Texaflam XPE foam coating were also the worst besides the untreated samples. Coating with Texaflam XPE ECO (SB/Br-free) was the best because the samples were partially burned in both 65% PES/35% CO and 100% polyester twill weaves. Especially when using energy of lower intensity, it's the structure of the sample that was destroyed changing the sample to yellow color. Long wavelength radiation gives rise to pyrolytic processes which involve heating.

This can be used for melting and hence for welding, or at higher intensity for vaporization or thermal degradation.

Table 7.1 Results of laser intensity on 100% polyester twill weave

Laser Intensity	Impregnation or coating used	Diameter		
		Diameter I	Diameter II	Average Diameter
High Intensity	Original	Burn out	Burn out	Burn out
	200gl^{-1}Texaflam PE	25mm	15mm	20mm
	500gl^{-1}Texaflam BS	26mm	12mm	19mm
	250gl^{-1}Texaflam XPE	35mm	25mm	30mm
	500gl^{-1}Texaflam XPE	36mm	20mm	28mm
	Texaflam XPE ECO 1x	15mm	15mm	15mm
	Texaflam XPE ECO 2x	17mm	13mm	15mm
Low Intensity	Original	Burn out	Burn out	Burn out
	200gl^{-1}Texaflam PE	12mm	18mm	15mm
	500gl^{-1}Texaflam BS	12mm	18mm	15mm
	250gl^{-1}Texaflam XPE	25mm	15mm	20mm
	500gl^{-1}Texaflam XPE	21mm	19mm	20mm
	Texaflam XPE ECO 1x	5mm	5mm	5mm
	Texaflam XPE ECO 2x	0mm	0mm	0mm

Table 7.2 Results of laser intensity on 65% polyester/ 35% cotton twill weave

Laser Intensity	Impregnation or coating used	Diameter		
		Diameter I	Diameter II	Average Diameter
High Intensity	Original	Burn out	Burn out	Burn out
	200gl^{-1}Texaflam PE	25mm	15mm	20mm
	500gl^{-1}Texaflam BS	26mm	12mm	19mm
	250gl^{-1}Texaflam XPE	35mm	25mm	30mm
	500gl^{-1}Texaflam XPE	36mm	26mm	31mm
	Texaflam XPE ECO 1x	10mm	10mm	10mm

	Texaflam XPE ECO 2x	10mm	8mm	9mm
Low Intensity	Original	Burn out	Burn out	Burn out
	200gl^{-1}Texaflam PE	10mm	10mm	10mm
	500gl^{-1}Texaflam BS	10mm	10mm	10mm
	250gl^{-1}Texaflam XPE	25mm	15mm	20mm
	500gl^{-1}Texaflam XPE	25mm	15mm	20mm
	Texaflam XPE ECO 1x	0mm	0mm	0mm
	Texaflam XPE ECO 2x	0mm	0mm	0mm

Samples of 100% polyester twill weave

Fig. 7.2 100% polyester twill weave original

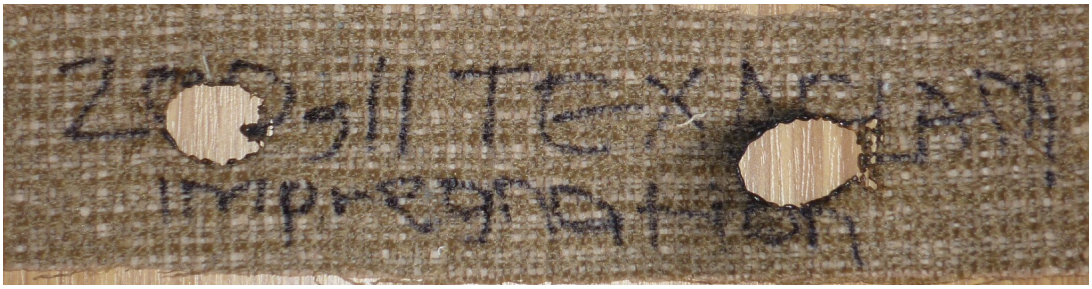


Fig. 7.3 100% polyester twill weave impregnated with 200g/l Texaflam Concentrated

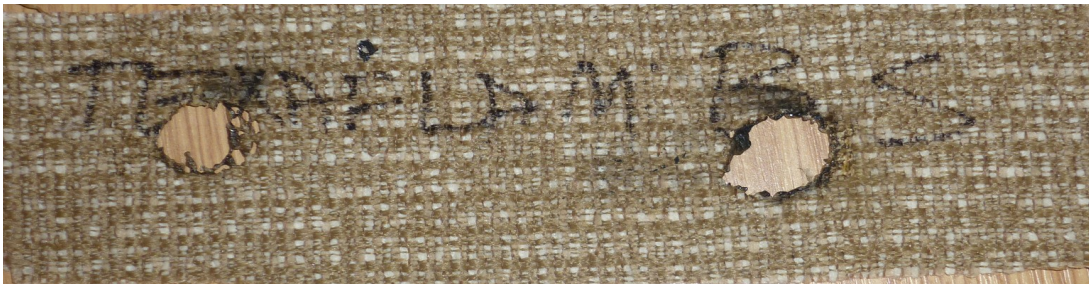


Fig. 7.4 100% polyester twill weave impregnated with 500g/l Texaflam BS

Fig. 7.5 100% polyester twill weave coated with 250g/l Texaflam XPE (SB/Br) foam coating

Fig. 7.6 100% polyester twill weave coated with 500g/l Texaflam XPE (SB/Br) foam coating

Fig. 7.7 100% polyester twill weave coated with Texaflam XPE (SB/Br) coating 1x

Fig. 7.8 100% polyester twill weave coated with Texaflam XPE (SB/Br) coating 2x

Samples of 65% PES /35% CO twill weave

Fig. 7.9 65% PES /35% CO twill weave original

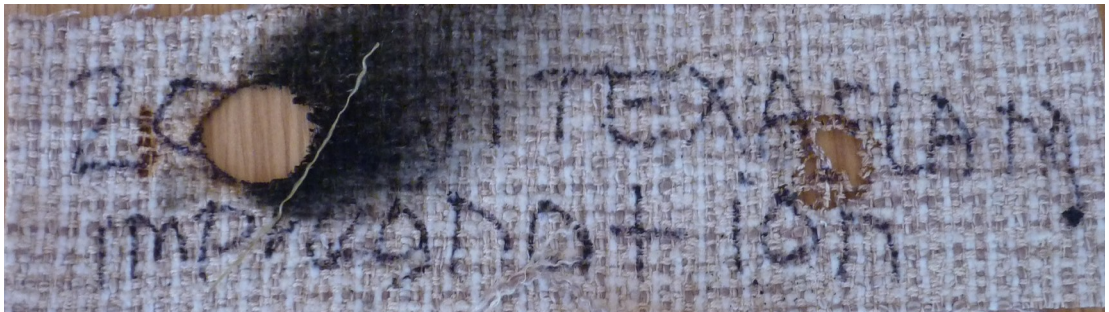


Fig. 7.10 65% PES /35% CO twill weave impregnated with 200g/l Texaflam Concentrated

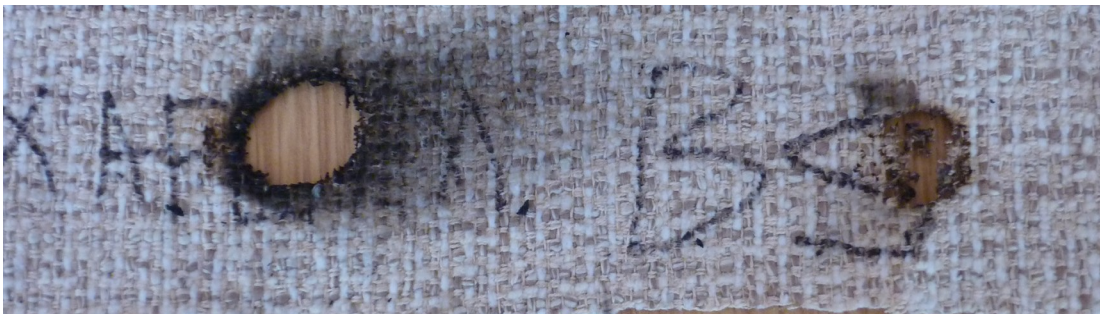


Fig. 7.11 65% PES /35% CO twill weave impregnated with 500g/l Texaflam BS

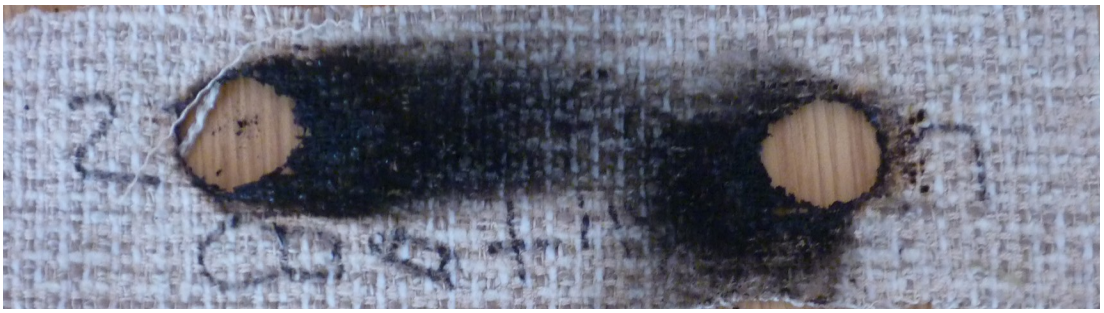


Fig. 7.12 65% PES /35% CO twill weave coated with 250g/l Texaflam XPE (SB/Br) foam coating

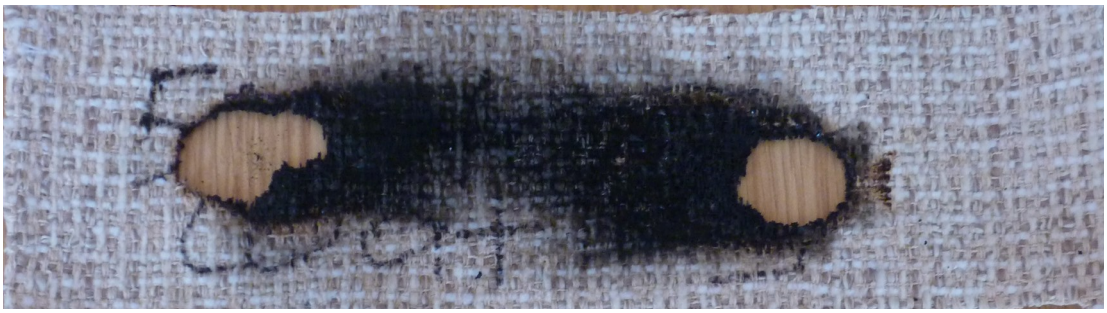


Fig. 7.13 65% PES /35% CO twill weave coated with 500g/l Texaflam XPE (SB/Br) foam coating

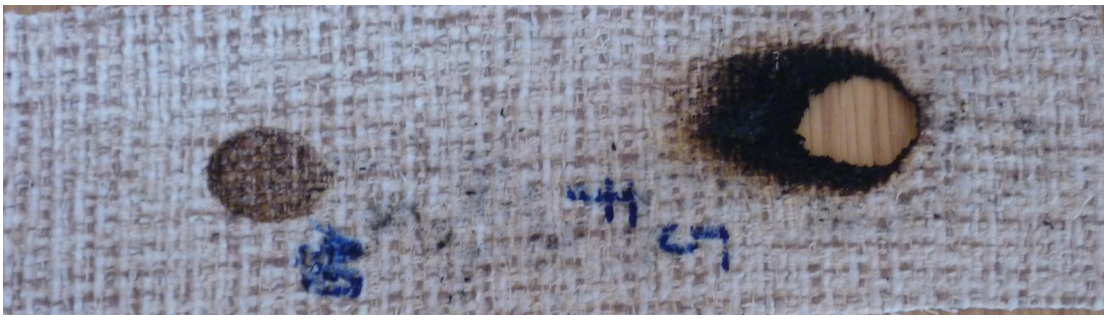


Fig. 7.14 65% PES /35% CO twill weave coated with Texaflam XPE (SB/Br) coating 1 x

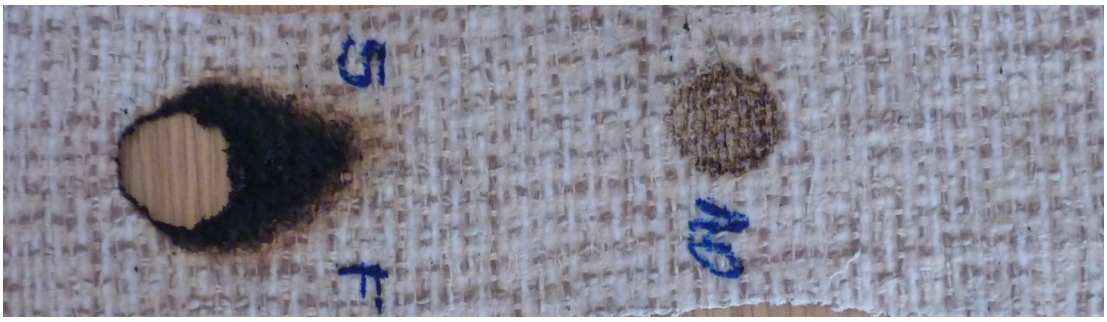


Fig. 7.15 65% PES /35% CO twill weave coated with Texaflam XPE (SB/Br) coating 2 xs

8 Conclusion

When samples were tested according to BS EN 1021–1/1021-2, the sample meets the requirements (smouldering cigarette and match Equivalent) pass. Samples used were pretreated. All samples, the coated and impregnated samples did not ignite when subjected to flame and all the untreated samples burned when exposed to flame. 100% polyester twill weave impregnated with 200gl^{-l} Texaflam PE Concentrated had good results than all textile used, it can be seen by looking at limiting oxygen index of 34.0% warp and 30.8% weft followed by same concentration on 65% PES/ 35% cotton twill weave and gave limiting oxygen index of warp 29.2% and weft 27.3%. Coating twice makes the textile to be more fireproof but less air permeable. Coating

twice with Texaflam XPE ECO on 100% polyester twill weave gave the higher pickup than 65% PES/ 35% cotton twill weave which was 28.82% and that made polyester to have low water vapor permeability and blended polyester-cotton with higher water vapor permeability. Coated textiles allow water vapour to be transported through hydrophilic parts of the polymer chains. Results are showing that, water proofness and breathability depended on the concentration of the paste and the structure of the textile. Again coating with foam gave higher limiting oxygen index on 100% polyester twill weave than 65% PES/ 35% cotton twill weave proving be the best textile in many cases.

Texaflam PE Concentrated is considered to have the better results looking at liming oxygen index results. The mixture of phosphonate complexes imparting self extinguishing, ability to polymeric materials when subjected to burning process. For the safety of environment, low add-ons of FR chemicals on to the substrate are needed from the ecological point of view and also for the commercial consideration. Foam coating, Texaflam XPE was very good on 100% polyester twill weave, foam still visible on the textile after drying. When exposed to fire polymer melt thickens and solidifies producing structure that prevented volatile liquids into the flame ensuring the limitations of heat transfer between the flame and the substrate. The experiment corresponded with theory. Flame retardants can reduce the heat release rate and prolong the time of heat release and heat release rate is the fundamental fire property of material.

Texaflam XPE ECO coatings were much better on blended 65% PES/ 35% cotton twill weave. $200gl^{-1}$ Texaflam PE Concentrated which was impregnated on 100% polyester twill weave decrease the lightness of the textile but with negligible difference on 65% PES/ 35% cotton twill weave. Also $500gl^{-1}$ Texaflam BS and coating twice with Texaflam XPE ECO made 65% PES/ 35% cotton twill weave to be darker. $200gl^{-1}$ Texaflam PE Concentrated is showing high proofness of fire and at the same time changing the color of polyester and also has total color difference of 4.69 higher. Also it can be concluded that when textiles are coated or impregnated by fire proof agents they change color. Coated twice of polyester-cotton blends have higher tensile strength than pure polyester. Coating created an adhesion between the coating textile used by penetrating into the weave structure and decrease further tearing. The penetration depended on the structure paste viscosity and method used.

Heating of textiles using DSC showed the melting of polyester and its blends around same temperature 260ºC. But impregnation with 500g/l Texaflam BS on 100% polyester twill weave caused a reaction which can be seen in fig 6.5, reaction of chemical reagent used in the textile. Resulting in melting from 122 to 227ºC with small peak of 180ºC and also the same reaction can be seen on 65% PES/ 35% cotton twill weave, where it started to melt at 138 to 232ºC . All the textiles used starts to recrystallize at 200ºC.

In all 65% PES /35% CO twill weave as soon as temperature starts rising, the samples began to melt because they contained trehalulose. The study of the thermal properties shows that trehalulose has the lowest melting point (48ºC). In addition, trehalulose is highly hygroscopic.

Laser treatment completely burns the textile especially when power is increase. That can be observed in fig 7.2 and 7.9 where original textiles burned without stopping. Most of the textiles were burned to an oval shape. Increase in energy, the laser beam is not so strong but increases in power the laser beam were much stronger. Coating with Texaflam XPE ECO when the energy was at 10J destroyed only the structure of the textile especially on textile that has been coated twice.

9 References

1. Subcommittee on Flame-Retardant Chemicals, Committee on Toxicology, Board on Environmental Studies and Toxicology, National Research Council.: Toxicological risk of selected flame retardant chemicals, National Academy of Science, 2000 [18 December 2009]
2. Jiri George Drobny.: Handbook of Thermoplastic Elastomers, William Andrew Inc 20007
3. S.M. Lomakin and G.E. Zaikov .: Ecological Aspects of Polymer Flame Retardancy, VSP - An imprint of BRILL, 1999

4. Barry Leonard.: Furniture Flame Retardancy Partnership: Environmental Profiles of Chemical Flame-Retardant Alternatives for Low-Density Polyurethane Foam, DIANE Publishing, 2005

5. Royale S. Underhill John Hiltz.: A Discussion of Polymeric Materials for Fire-Safe Naval Applications Enhanced Reliability, Fire and Damage Tolerance, Defence R&D Canada – Atlantic Howard Moyst Advanced Industrial & Mechanical (AIM) Engineering, August 2007 [10 February 2010]

6. Pietro Bellini, Ferruccio Bonetti, Ester Franzetti, Giuseppe Rosace, Sergio Vago.: Reference Book of Finishing, ACIMIT Foundation, 2001

7. Darnerud, S. Atuma, M. Aune, W. Becker, R. Bjerselius, S. C nattingius and A. Glynn.: "New Thinking on Flame Retardants, Environmental Health Perspectives", 116(5), May 2008 "Polybrominated diphenyl ethers in breast milk from Uppsala County, Sweden." Environmental Research 93(2): 186–194. PMID 12963403 by Y. Lind, P. O., 2003 [24 February 2010]

8. Charles E. Wilkes, James W. Summers, Charles A. Daniels.: PVC Handbook, Carl Hanser Verlag, 2005

9. Derek Heywood Textile and chemical consultant, Sacola Associates "Textile Finishing", Cheadle Hulme, UK; Finishing by A. R. Horrocks, "Flame-retardant Finishes", Flameretardants.pdf - Search flame retardants [26 March 2010]

10. Levchik, Sergei and Wilkie, Charles A..: Char Formation, Ch. 6, pp. 171–216, In Grand and Wilkie, 2000

11. A.R. Horrocks and D. Price.: Fire retardant materials, Woodhead Publishing Limited, 2001

12. James G. Cook.: Handbook of Textile Fibres Vol. I. Natural Fibres.: Merrow Publishing CO. LTD, 1984

13. Phillip J. Wakelyn, Noelie R. Bertoniere, Devron P. Thibodeaux, Barbara A. Triplett, Alfred D. French.: Cotton Chemistry and Technology, Taylor and Francis Group, LCC, 2007

14. Menachem Lewin, Stephen B. Sello.: Handbook of Fiber Science and Technology: Chemical Processing of Fibers and Fabrics: Fundamentals and Preparation, Vol. 1, Library of Congress Cataloging in Publication Data, 2001

15. B Mahltig.: Nanosols And Textiles, World Scientific Publishing Co. Pte. Ltd. 2008

16. Roberto A. Lopez-Anido, Tarun R. Naik, Gary T. Fry, David A. Lange (Editor), Vistasp M. Karbhari.: Emerging Materials for Civil Infrastructure: State of the Art, Library of Congress Cataloging In Publication Data, 2000

17. Charles A. Harper, Edward M. Petrie.: Plastic materials and processes, John Wiley and Sons Inc., 2003

18. A.R. Horrocks and S.C. Anand.: Handbook of Technical Textiles, Woodhead Publishing Limited, 2000

19. Johnson J R and Sujarit C.: "Flame resistant polyester/cotton fabric and process for its production", US Patent 4,748,705, assigned to Burlington Industries, 1988

20. W.D. Schindler, P.J. Hauser.: Chemical Finishes of Textiles, Woodhead Publishing Limited, 2004

21. Hauser P J, Triplett B L and Sujarit C.: Flame-resistant cotton blend fabrics', US Patent 4,732,789, assigned to Burlington Industries, 1988 [18 December 2009]

22. http://www.cefic-efra.org/Objects/2/Files/OtherPhosphorusFactSheet.pdf [Dr B. Dero.: European Flame Retardant Association 30 March 2010]

23. Charles A. Wilkie, "An Introduction to the Use of Fillers and Nanocomposites in Fire Retardancy" Department of Chemistry, Marquette University, PO Box 1881, Milwaukee, WI 53201, U.S.A. [18 December 2009]

24. J. Innes, Innes.: A. Plastic flame retardants: technology and current developments, Rapra Technology Limited, 2003

25. Hans-Joachim Streitberger and Karl-Friedrich Dossel.: Automotive Paints and Coatings, Second completely revised and extended edition, WILEY-VCH, 2008

26. A.K. Sen.: Coated Textile: Principle and Applications, Technomic Publishing Company, Inc, 2001

27. Joel S. Miller, Marc Drillon.: Magnetism: molecules to materials, Wiley-VCH, 2001

28. Roger P. Brown.: Handbook of polymer testing: physical method, Library of Congress Cataloging-in-Publication Data, 1999

29. L. A. Utracki.: Polymer blends handbook; Kluwer academic publishers, 2002

30. D. Satas, Arthur A. Tracton.: Coating Technology Handbook, Second edition revised and expanded, Marcel Dekker Inc, 2001

31. Walter Fung.: Coated and Laminated Textiles, Woodhead Publishing Limited in Association with the Textile Institute, 2002

32. Walter Fung, Mike Hardcastle.: Textile Institute Textiles in Automotive Engineering Technology & Engineering, Woodhead Publishing Ltd, 2001

33. J.V. Koleske.: Paint and Coating Testing Manual, Library of Congress Cataloging-in-Publication Data, 1995

34. Johannes Karl Fink.: Reactive Polymers Fundamentals and Applications, William Andrew Inc, 2005

35. Arthur A. Tracton.: Coating Technology Handbook, Third Edition, Taylor & Francis Group LLC, 2006.

36. Ulrich Meier-Westhues.: Polyurethanes Coatings, Adhesives and Sealants, Vincentz Network GmbH & Co. KG. Hannover, 2007

37. Michael Szycher.: Szycher Handbook of polyurethanes, CRC Press LLC, 1999

38. Carlos J. Hilado.: Flammability Handbook for Plastics, Fifth Edition, Technomic Publishing Company Inc, 1998

39. G.W.H. Höhne, W.F. Hemminger, H. J. Flammersheim.: Different Scanning Calorimetry; Second Edition, Springer-Verlag Berlin Heidelberg, 2003
40. Marcel Mulder.: Basic Principles of Membrane Technology second edition, Kluwer Academic Publishers, 1996

41. David Sheehan.: Physical Biochemistry: Principles and Applications, John Wiley & Sons Ltd, 2009

42. János Schanda.: Colorimetry: understanding the CIE system, John Wiley & Sons, 2007

43. http://www.gamutvision.com/docs/printest.html [Digitization and Metric Conversion for Image Quality Test Targets by William Kress 16 April 2010]